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TECHNICAL CONFERENCE ON WOOL FABRIC FINISHING

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Held at Albany, California
February 9-10, 1961

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THE TECHNICAL CONFERENCE ON WOOL FINISHING reported here was held February 9 and 10, 1961, in the Western Regional Research Laboratory, Albany 10, Calif. This Laboratory is headquarters of the Western Utilization Research and Development Division of the Agricultural Research Service, U. S. Department of Agriculture.

One of the units of this Western Division is the Wool and Mohair Laboratory, whose staff consists of 40 chemists, physicists, fiber technologists, and engineers. Their research program has been enlarged in recent years. In 1959 their facilities were extended to include equipment for processing wool from raw fleece to finished worsted.

The meeting was attended by representatives of industrial and governmental groups concerned with fiber research and development, wool growers' associations, the wool processing and finishing industries, and chemical manufacturers. Staff members of the Wool and Mohair Laboratory reported recent researches, particularly on new finishes for wool fabrics. Representatives of industrial andother groups discussed problems and progress in the broad field of new finishes for apparel wool. Progress in chemical finishing of cotton was discussed by a representative of the Southern Utilization Research and Development Division of the U. S. Department of Agriculture. The reports presented and the discussions that followed are included in this report.

The program was developed by M. J. Copley, Director of the Western Division and H. P. Lundgren, Chief of the Wool and Mohair Laboratory, in cooperation with other staff members and with advisers in other governmental agencies and industry.

This report was prepared in the Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Albany 10, Calif. Copies are available on request.

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WOOL FINISHING RESEARCH AT THE WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

Harold P. Lundgren
Western Regional Research Laboratory
USDA, Albany, California

Recent progress in wool textile finishing research in this Division warrants discussion, so that it can be understood in detail by interested representatives of the wool industry. By discussion we hope to determine what further development may be needed to make these results practically useful. We want to encourage industry to try out these new processes. In addition, we have planned more general discussion of finishing requirements partly to suggest new useful research objectives.

Research on the utilization of wool and mohair began here in 1948. The program was greatly expanded in 1959 with construction of the Wool Processing Laboratory. This has proved immensely valuable. Our applied research and development are now centered in the new Laboratory, which is equipped with machinery for studying wool processing by the worsted system from opening, dusting, and scouring greasy wool, through carding, combing, spinning, weaving, and the final series of finishing operations. With these facilities we can now undertake to translate the more promising laboratory findings into practical use.

Finishing is the series of processes following weaving. The result is cloth that is ready for garment making or comparable end use. Similarly, finishing also denotes the series of processes given made-up garments or other textile products after the principal manufacturing steps in order to achieve attractive, salable products.

Certain finishing operations (fulling, steaming, pressing, shearing, and drying) are primarily mechanical. In chemical finishing, chemicals are applied to the fabric to impart or enhance desired properties. After application, the substances added may in some cases undergo further reaction. The objectives are improvements in such properties as softness, resistance to soiling, repellency to water or oil, reduction of drying time, resistance to wrinkling, resistance to pilling, and stability of color. Improvement of ease of care is of special concern in research here.

Easy care demands ultimately that presentable appearance be maintained with minimal effort. It suggests garments that can be machine-laundered at home, tumble-dried or drip-dried, and then worn without ironing or other care, retaining a neat, new appearance after repeated use. To achieve such a utopian result, the ideal finishing process must impart durable resistance not only to felting shrinkage, but also to mussing, fuzzing, and pilling. Pleats and creases must stay sharp. On the other hand, the finish must not impair the desirable qualities of wool's hand and appearance. Ideally, there must be no loss of softness or liveliness, no decrease in strength or wear-life, no development of off-odor, no impairment of color or color stability. Above all, the treatment must be economical and be easy to apply with existing mill equipment. These are stringent requirements. We believe that progress toward these

goals justifies presentation and detailed discussion of our results. This view is encouraged by many favorable comments from mills and finishers for whom samples have been treated.

But even when a finish is achieved that performs well in laboratory tests, it must still pass the test of performance as a completed garment. The new finishes have not had thorough trial in consumer goods. Wear tests are now being planned. The few garments made so far disclose no problems, provided shrink-resistant linings and sewing thread are used. The new finishes have been applied to many dyed fabrics without difficulty, but shade changes have occurred in a few instances. Resin-treated fabrics have been dyed without crocking or difficulty in achieving levelness.

By presenting details of results so far achieved, we hope to hasten appropriate commercial application and further progress.

EPOXY-AMINOPOLYAMIDE RESIN FINISHES TO STABILIZE WOOL AGAINST FELTING SHRINKAGE

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This summary describes the basis for the epoxy-aminopolyamide treatment and recent experimental results. Suggested recipes for practical trial are available on request. Earlier work is described in two papers: "Prevention of Wool Fabric Shrinkage with Polyamide-Epoxide Resin Finishes," C. E. Pardo and R. A. O'Connell, Amer. Dyestuff Reptr. 47: 333-8 (1958) and "Improvements in Epoxy-Aminopolyamide Finishes for Easy-Care Wool Fabrics," C. E. Pardo, Jour. Textile Inst. 51 (12, part II): T1462-73 (1960). Patent applications have been filed covering various aspects of these and related treatments.

Factors governing felting shrinkage and shrinkage control. The properties of wool mainly responsible for felting shrinkage are its surface scale structure, which helps the fiber to move more easily in one direction than the other, its plasticity when wet, and its long-range elasticity. Treatments to prevent shrinkage usually depend either on eliminating the differential frictional effect by removing or covering the scale surface, or on bonding fibers together to hinder relative movement. More subtle effects may also be important.

Resin finishes made from blends of epoxy and aminopolyamide resins that have been developed here successfully limit felting shrinkage under certain conditions. Commercial development hinges on adequate control of processing requirements, on achieving an acceptable compromise between shrinkage control and alteration of hand or color for a given fabric, and on economic factors.

Chemistry and technology of epoxy-aminopolyamide finishing. The value of epoxy resins for wool finishing is suggested by their commercial availability at reasonable cost and their chemical stability, adhesion and durability when cured. Use of the aminopolyamide curing agent permits good control of properties of the cured resin such as flexibility. Structures and reactions of these components are illustrated in Figure 1. Linear or cross-linked resins form, depending on the number and position of reactive sites in the reacting molecules. The greater the number of reactive sites on each reagent, the less soluble, less flexible, and more adherent the coating. Reaction of the epoxide component with amino, hydroxyl, and carboxyl groups of the wool proteins provides means of durably bonding the resin finish to the wool protein.

Two types of commercially available epoxide resins were studied in work here. One is a condensation product of epichlorhydrin with bisphenol A to give an epoxide equivalent of 175 to 210; the second is a condensation product of epichlorhydrin and glycerol to give an epoxide equivalent of 140 to 165. (The epoxide equivalent is the number of grams of the resin per gram atom of reactive epoxide oxygen.) The amino polyamide resins are commercially available condensation products of polyamines and heat-dimerized fatty acids. Use of excess polyamine yields polymers with reactive amino groups available for

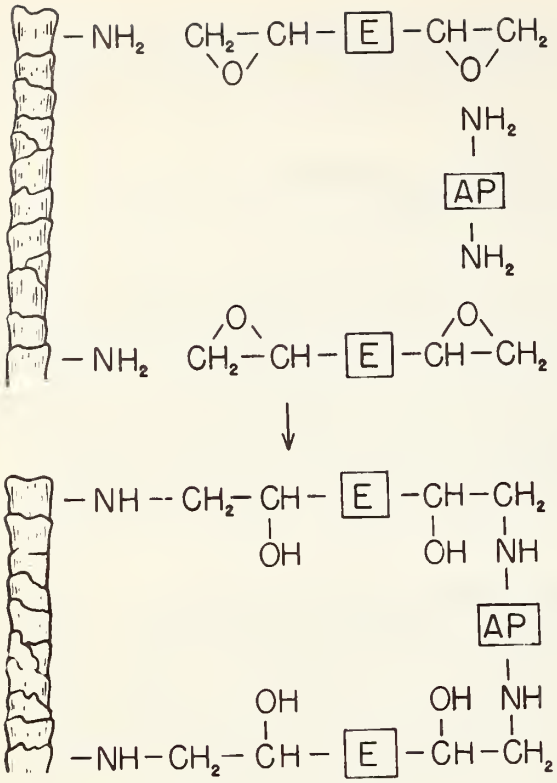

Figure 1. Reaction of epoxy and aminopolyamide resin components with wool proteins. Unreacted constituents are shown above, with only atoms of reactive groups shown explicitly. The wool fiber is at the left with two primary amino groups as examples of reactive sites indicated. The epoxy resin is designated E with two (of many) epoxide groups. The aminopolyamide resin is designated AP with two (of many) amino groups. On reaction a cross-linked network is formed, presumably directly bonded to the wool protein, as suggested at the bottom of the diagram.

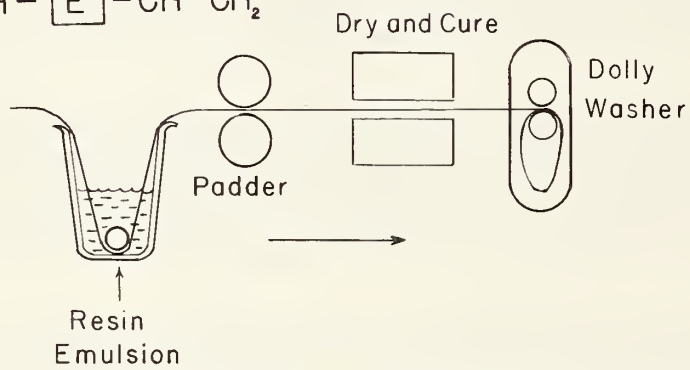

Figure 2. Sequence in applying epoxy-aminopolyamide resin to fabric by padding. Fabric passes through resin emulsion at left and through squeeze rolls. It is then dried, cured, dolly-washed, and finished conventionally.

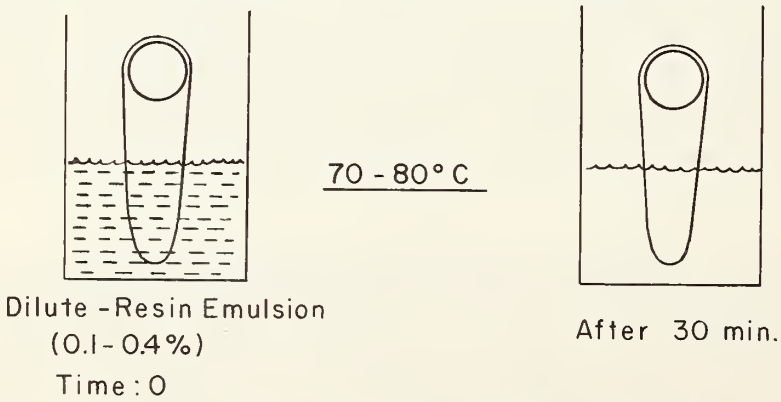

Figure 3. Application of epoxy-aminopolyamide resin by exhaustion. Fabric as endless belt is circulated through dilute resin emulsion until most is taken up. Fabric can then be dyed and precured in the same equipment.

reaction with the epoxy component. The resins are applied to fabric as a mixed emulsion in either of two ways, by padding or by exhaustion. The pad method, using conventional equipment, is diagramed in Figure 2. The fabric is dipped into the emulsion, run full width through squeeze rolls into a drying and curing oven, and then dolly-washed before final finishing such as dyeing, napping, pressing, or decating. In the exhaustion method (Figure 3), the resin components are taken up by the fabric in much the same way as dye is taken up in piece dyeing. The same equipment is used and the procedure is similar.

Preparation of resin emulsions. To prepare a resin dispersion for padding, the required amounts of the epoxy and aminopolyamide components are separately thinned with solvent, typically ethyl acetate or an aromatic hydrocarbon product with relatively high flash point. A nonionic emulsifying agent is incorporated into the epoxy solution in an amount equal to 5% of the combined weight of the resins. Water required is acidified with 1.5 to 2.5 milliequivalents of acetic acid (0.9 to 1.5 grams, glacial) per gram of the aminopolyamide resin used, so that the resulting dispersion will have a pH between 8.5 and 9.0. The weight ratio of epoxy resin to aminopolyamide is chosen to be between 60:40 and 70:30. The thinned resin solutions are mixed and then stirred into part of the acidified water to give a 10 to 20% emulsion. This emulsion is then passed through a colloid mill or other effective emulsifier and finally diluted with the rest of the water to the desired concentration. The resin particle sizes range from less than one micron up to three microns.

Alternative emulsifying procedures have been studied, notably by Charles H. Binkley of this laboratory, who has achieved emulsions with submicroscopic particle sizes. The requirements for acceptable emulsification are (1) that acid be provided to neutralize the aminopolyamide resin and (2) that a high-speed mixing device be used when the combined resin solution is added to the water. Two exceptions have been noted. First, no acid is needed if ethyl acetate is used as the initial solvent for the resins. Second, when isopropyl alcohol is used as solvent for the resins (and only if the epoxy component is based on glycerol rather than on bisphenol A), clear colloid-like dispersions are formed when the resin solution is stirred by hand into acidified water.

Applying resin by padding. Before applying resin, it is necessary to give the fabric a mild alkaline scour to remove residual oil and the like and to insure that the fabric (especially if dyed) is neutral or slightly alkaline. A suitable scouring bath uses 0.2 to 0.8% tetrasodium pyrophosphate with 0.05 to 0.1% of a nonionic wetting agent. The goods are scoured for a half hour at 120 to 130° F., rinsed, and dried. To apply the resin, the dry fabric is run at full width through the resin emulsion and then through squeeze rolls, with roll pressure adjusted to give 50 to 70% wet pickup. The amount of resin applied is controlled by the concentration of the emulsion and to a lesser extent by the roll pressure. The fabric then goes directly to the drying oven, where, depending on the heat exchange capacity and speed, drying and curing can be accomplished in one pass. An afterwash in a dolly washer is used to remove unreacted resin and to soften the fabric.

The temperature and time of drying and curing can be varied widely, depending on the interval between curing and dolly-washing. For example, the resin can be dried and cured at 275 to 300°F. in 5 to 10 minutes. Fabrics cured under these conditions can be dolly-washed at once. However, one may also take advantage of the possibility of curing bisphenol A-based epoxy-aminopolyamide systems at room temperature. In this case lower drying temperatures can be used if enough time elapses before washing. For example instead of curing at 275°F., one can cure at 225°F. for 10 to 15 minutes, or at 250°F. for 5 to 10 minutes provided the fabric is then kept at room temperature for at least 24 hours before washing.

Dolly washing is adjusted in time and severity according to the fabric construction and the amount and formulation of the resin. Typical conditions are 120°F. with 0.1% nonionic detergent. After washing, the fabric is rinsed with slightly acidified water sufficient to bring the fabric to neutrality or slight acidity. The hand can be further improved by adding 0.25 to 0.5% of a conventional softener to the fabric. This is accomplished by padding just before drying. The softener may also be applied by exhaustion in the dolly washer.

Applying resin by exhaustion. Emulsions are prepared for application by exhaustion just as for padding except that they are diluted to 0.1 to 0.4% resin solids and are acidified with sulfuric acid to pH 7.0 to 7.5. Prepared fabric is entered into the emulsion in a dye beck and exhaustion carried out typically for an hour at 160 to 175°F. The material is then rinsed and dyed, if desired, or softened and finished as with the padding method. This method is still undergoing active development.

Testing methods. Three sorts of shrinkage tests are used routinely. Experimental finishing treatments, with their numerous variations, are first screened by a two-minute accelerated wash test with paired 6x5-inch (warp x fill) swatches in an Accelerotor ("The Accelerotor for Abrasion Testing and Other Purposes," H. W. Steigler, H. E. Glidden, G. J. Mandikos, and G. R. Thompson, Amer. Dyestuff Reptr. 45: P685, 1956). Edges of the cloth are bound with rubber cement to prevent ravelling. Before the test the cloth is wet out and conditioned in 1% sodium acetate for one-half hour at 120°F. and known distances laid out along warp and fill. Paired swatches are then washed two minutes in 200 ml. of 0.5% sodium oleate at 105°F. To make the treatment more reproducible, the variable speed motor of the original instrument is replaced by a 1/3 h.p. synchronous motor and the washing chamber is preheated with hot water. After the wash, the swatches are rinsed, the water squeezed out by hand, and measurements of warp and fill shrinkage made while the fabric is still damp.

Second, a 75-minute accelerated wash test at 105°F. is made in a reversing agitator-type home washing machine with a three-pound ballast load. This is a modification of the AATCC test, which uses a rotary washer. ("Tentative Test Method 99-1960: Relaxation and felting shrinkage of woven or knitted wool textiles," Technical Manual and Yearbook, 1960, Amer. Assoc. Textile Chemists and Colorists, Lowell, Mass.)

Finally, a multiple wash test is carried out in a home washing machine. Ten to 20 three-minute washings are carried out consecutively using the

"modern fabric" agitation cycle. The individual washes are sometimes followed by tumble-drying.

Comparison of shrinkage test methods. The Accelerotor wash test gives about the same area shrinkage on untreated fabric as does the 75-minute accelerated washing machine test. These are approximately equivalent to 15 wash-tumbledry cycles. Tensile strength, Elmendorf tear strength, and stiffness are determined by standard ASTM procedures. (Standard General Methods of Testing Woven Fabrics, ASTM Designation D 39-59, "Breaking Strength by Cut Strip Method," page 4; ASTM Designation D 1682-59T, "Tentative Methods of Test For Breaking Load and Elongation of Textile Fabrics," page 740; ASTM Designation D 1424-59, "Standard Method of Test for Tear Resistance of Woven Fabrics by the Falling Pendulum (Elmendorf) Apparatus" (except with 6400 gram augmenting weight), page 607; ASTM Designation D 1388-55T, "Tentative Methods of Test for Stiffness of Fabrics, Method A. Cantilever Test," page 591. ASTM Standards on Textile Materials, Committee D-13, 1959. American Society for Testing Materials, Philadelphia, Penn.)

Results and evaluation. A yellowing tendency appears with aminopolyamide plus glycerol-based epoxy resin cured at high temperature. Yellowing is lessened by incorporation of bisphenol A-based epoxy component. As the proportion of the bisphenol A component is increased (for a given resin add-on of 5%, based on initial dry weight), yellowing decreases and shrinkage protection improves, but the stiffness also increases. Details are shown in Figures 4 and 5. These show also the effects of various preliminary treatments, including use of hydrogen peroxide, which is especially valuable with formulations with high proportions of glycerol-based epoxy resin. The data on stiffness emphasize the need for use of no more resin than is required for stabilization.

Dolly washing after the resin is cured improves the hand, but the time needed must be economically reasonable. Other methods of mechanical conditioning may need to be developed. Washing time required for effective softening also depends on the resin formulation as shown in Figure 6. For example, a fabric finished with 7% resin consisting of 40 parts (by weight) of glycerol-based epoxy component, 10 parts of bisphenol-based epoxy, and 50 parts of aminopolyamide reaches the same softness in 20 cycles in the dolly washer as the same fabric with 1.9% resin based on equal parts of bisphenol A and aminopolyamide and given 50 washing cycles. The same factors are illustrated again in Table 1, contrasting flexural rigidity of 6.9-ounce worsted flannel given various resin finishes with comparable resin uptake, adequate shrinkage protection, and little effect on tear strength or wrinkle recovery. Formulation of the resin with both epoxy components strikingly decreases rigidity in comparison with finishes using bisphenol A-based epoxy resin exclusively for the epoxy component.

Important differences among the formulations also appear in their keeping quality; that is, in the useful life of the emulsion after it has been prepared. When the epoxy component is exclusively based on bisphenol A, the resin must be applied within 6 to 8 hours for good results. Formulations with substantial proportions of glycerol-based epoxy component and in which

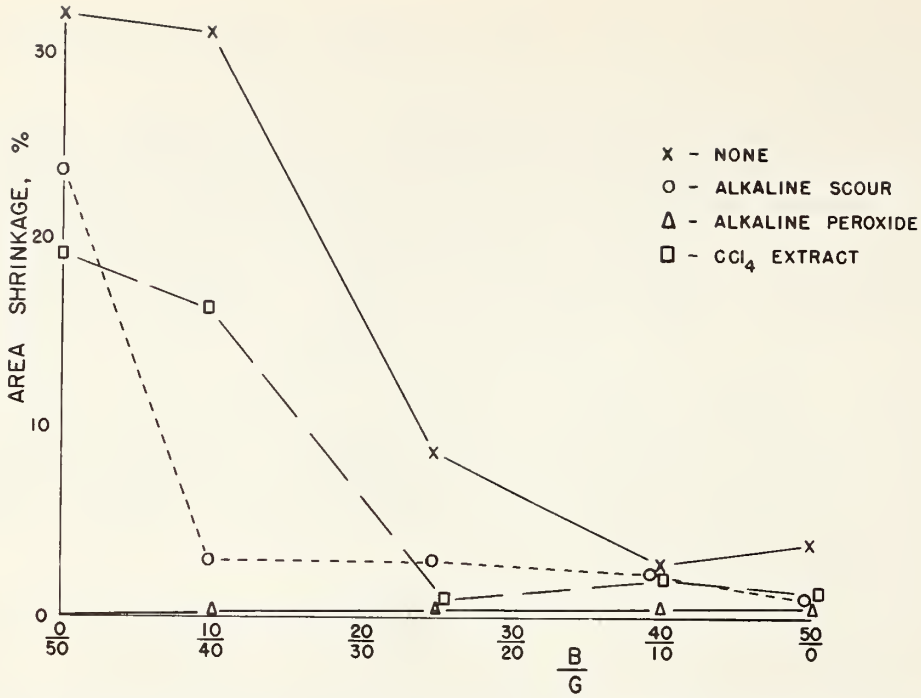

Figure 4. The shrinkage protection given by epoxy-aminopolyamide resin finishes is influenced by pretreatment and by the ratio of bisphenol A-based (B) and glycerol-based (G) epoxy components. Other conditions such as the resin uptake are comparable.

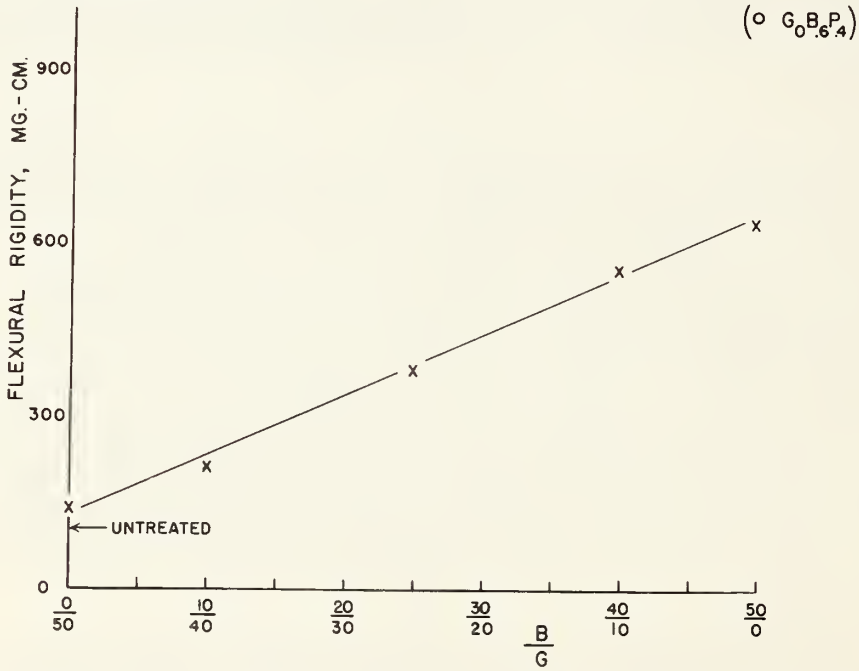

Figure 5. The stiffness, as flexural rigidity, of fabric given an epoxy-aminopolyamide finish, increases regularly with the ratio of bisphenol A-based (B) to glycerol-based (G) epoxy component (other conditions being comparable). The single point (upper right) shows the rigidity of fabric finished with no glycerol-based component. An acceptable balance must be found among yellowing, stiffness, and shrinkage control.

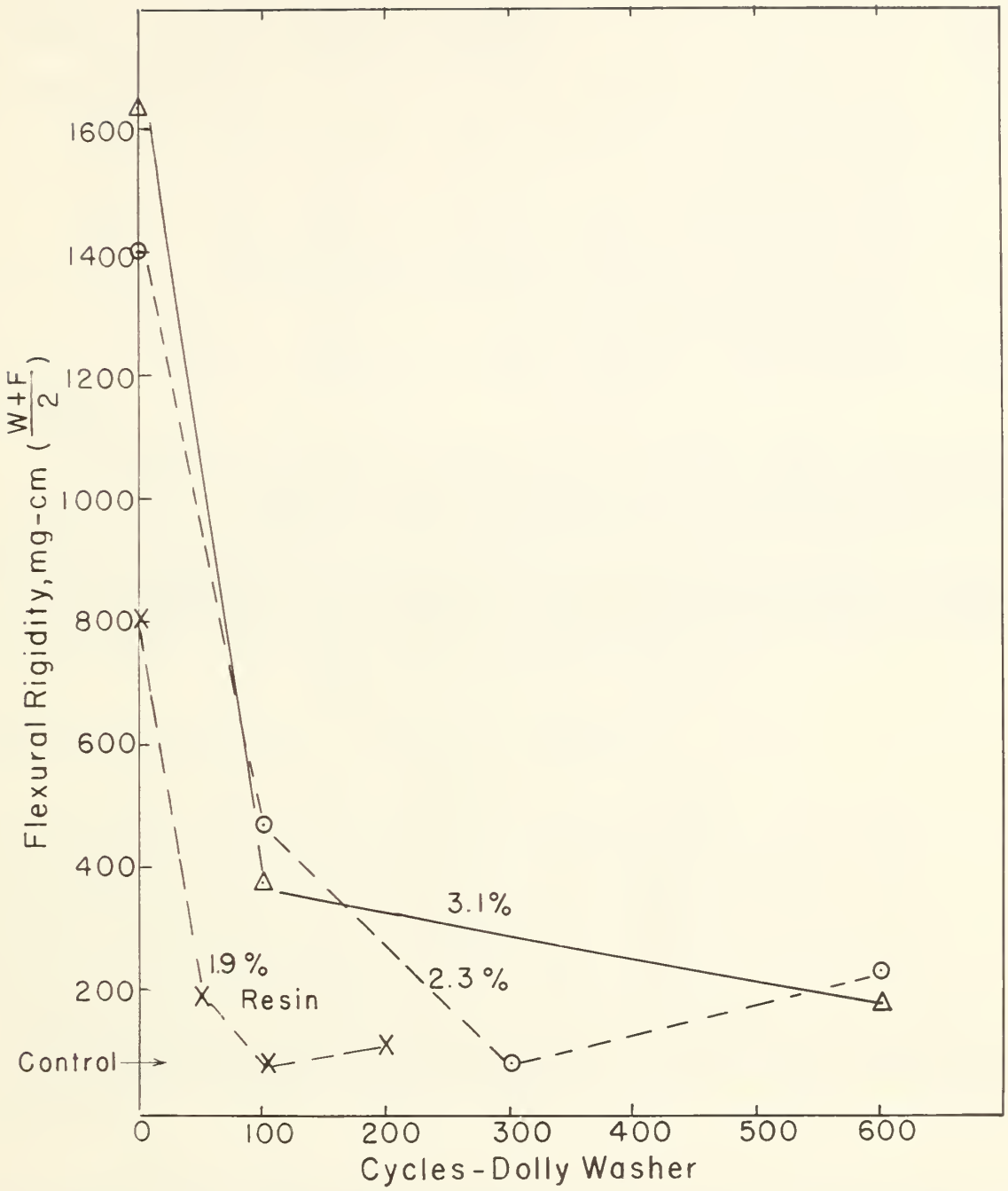

Figure 6. The stiffness of resin-finished fabrics can be greatly improved by dolly-washing after the treatment. This graph shows the average flexural rigidity in warp and fill directions of fabrics, with three levels of resin application, after dolly-washing for various times. Shrinkage protection is not impaired by this softening treatment.

Table 1. Physical properties of wool flannel treated with two epoxy-aminopolyamide formulations.

Resin composition G:B:P wt. %	Pretreatment	Uptake %	Area shrinkage (20 washes)	Tear strength (warp) g	Wrinkle recovery (warp) %	Flexural rigidity % increase
40:10:50	Alkaline scour	2.3	2.8	2000	83.6	43
	Alkaline peroxide	3.1	2.4	2140	83.8	61
0:60:40	Alkaline scour	3.6	4.8	1900	83.7	235
	Alkaline peroxide	5.1	2.0	1820	79.7	463
Control	Alkaline scour	none	70.5	2000	83.9	0 (reference)

G: glycerol-based epoxy component. B: bisphenol A-based epoxy component. P: amino polyamide component. Measurements of tear strength, wrinkle recovery, and rigidity were made on samples washed ten times.

Table 2. Effect of pH and age of resin dispersion on shrinkage.

pH	Age of resin emulsion, days 0	1	7
	Shrinkage in area, %		
4	25.6	14.4	43.8
6	4.9	0	26.9
8	3.0	4.0	1.0
10	3.0	3.0	2.0

The shrinkage is that produced by two Accelerotor washes. Resin formulation: 6% glycerol-based epoxy resin, 4% aminopolyamide resin, aged at room temperature.

isopropyl alcohol has been used to thin the resins initially (yielding a clear colloidal dispersion) have a much greater useful working life. The evidence is shown in Tables 2 and 3.

As a final example, Table 4 shows the possibility of getting good shrinkage control by the exhaustion method with very little change in hand. For the best results so far obtained, the resin is applied to undyed material pretreated with hydrogen peroxide. Exhaustion is not very efficient: only 20 to 30% of the resin is deposited. Although excellent results have been obtained on a limited range of material, technical problems in using

Table 3. Area shrinkage of fabric treated with resin dispersion and aged at 50°C., pH 8.3.

Aging time (hours) before application	0	1	3.5	27.5	100	108
Area shrinkage % (one Accelerotor wash)	0	0	0	1	0	2
(three Accelerotor washes)	1	0	2	1	10.7	26

Table 4. Shrinkage resistance of fabric treated with epoxy-aminopolyamide resin by exhaustion.

Fabric	Resin uptake %	Accelerotor	Accelerated (75-minute) washing machine test	Mild wash and tumble dry, 10 cycles	Flexural rigidity, % increase
Treated	0.1	2	2.4	3.9	2
Control	0	50	52	35	0

The resin formulation contained 40 parts glycerol-based epoxy component, 10 parts bisphenol A-based epoxy component, and 50 parts aminopolyamide component, applied as a 0.2% emulsion. The flexural rigidity was measured after washing 15 minutes at 38°C. in an automatic home washing machine.

exhaustion are still being overcome, so that it is premature to discuss this aspect in further detail.

Summary. Epoxy-aminopolyamide resin emulsions can be applied to a variety of wool fabrics, except (in the present state of the art) very fine soft materials or knit goods, to obtain consistent, durable shrinkage control with little impairment of hand or color. The chemical cost is 2 to 5 cents per pound of goods treated. Treatment, from an aqueous system, can be continuous. The treatment is additive; wool substance is not lost. With proper care, there is no significant loss in tensile or tear strength. The treatment is effective with resin add-on of 1.5 to 3% or 3 to 6%, governed by the respective choice of epoxy components based on bisphenol A or glycerol. The finish tends also to improve muss resistance. Resin does not deposit objectionably in the bath nor accumulate on squeeze rolls. There is no special odor problem.

On the other hand, the described special requirements must be met in making up the emulsions. If emulsification is not adequate, application is likely to be nonuniform, leading to local yellowing and nonuniform protection. A balanced formulation is needed to get the best compromise between protection, stiffness, and off-color of light shades. Stiffness may be critically dependent on care and skill in succeeding steps in finishing. Experience shows that technically satisfactory results can be obtained in many instances.

Discussion of Mr. Pardo's Report

Beaumont (referring to Table 2 showing apparent maximum shrinkage protection with a resin emulsion prepared at pH 6 and held for one day before use): Is the result of the pH 6 test reproducible?

Pardo: The trends shown are believed reliable. Individual shrinkage results may be in error by ± 2-1/2% (standard deviation for untreated fabric).

Wolff: What is the molecular size of the resins used?

Pardo: The commercial aminopolyamide resin is reported to have a molecular weight of about 6,000-10,000. The epoxy resin based on bisphenol A is reported to have an approximate molecular weight of 400; the one based on glycerol, approximately 300. These are the values before curing.

Wolff: What I was getting at is that certain low-molecular-weight epoxides are carcinogenic. Is this resin treatment likely to present a mill hazard?

Pardo: The uncured epoxy components (and especially that based on glycerol) are skin irritants and sometimes sensitizing agents. According to manufacturer's data and in our experience, ordinary care in handling the resins and uncured treated fabric, using disposable plastic gloves, is sufficient to avoid any immediate toxic symptoms. Possible long-range or cumulative effects of slight exposure have not been determined.

Wolff: Wool may be in a better position than cotton with respect to resin treatment. Cotton apparently needs to have the resin distributed throughout the fiber, requiring low-molecular-weight reagents. If wool can be usefully modified with a surface film, higher-molecular-weight reagents can be used. For this reason the toxicity hazard can be reduced.

POLYAMIDE SHRINK-RESIST FINISHES FOR WOOL BY INTERFACIAL POLYMERIZATION
I. LABORATORY DEVELOPMENT

Robert E. Whitfield
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USDA, Albany, California

The research discussed here is reported in more detail in "Wool Fabric Stabilization by Interfacial Polymerization, Part I, Polyamides," R. E. Whitfield, L. A. Miller, and W. L. Wasley, Textile Research Jour. (accepted for publication).

The chemistry of interfacial polymerization. Many condensation polymers can be made by interfacial polymerization (IFP), in which reagents in immiscible solvents react at the interface between the solvents. In particular, a water solution of a diamine, such as hexamethylene diamine, reacts almost instantaneously at its boundary of contact with a solution of a diacid chloride, such as sebacoyl chloride, in an inert, water-immiscible solvent, forming a film of polyamide which can be collected continuously. (This reaction was demonstrated.) The "polymerization" is based on the Schotten-Baumann reaction of an acid chloride with a compound containing active hydrogen, as shown in Figure 1.

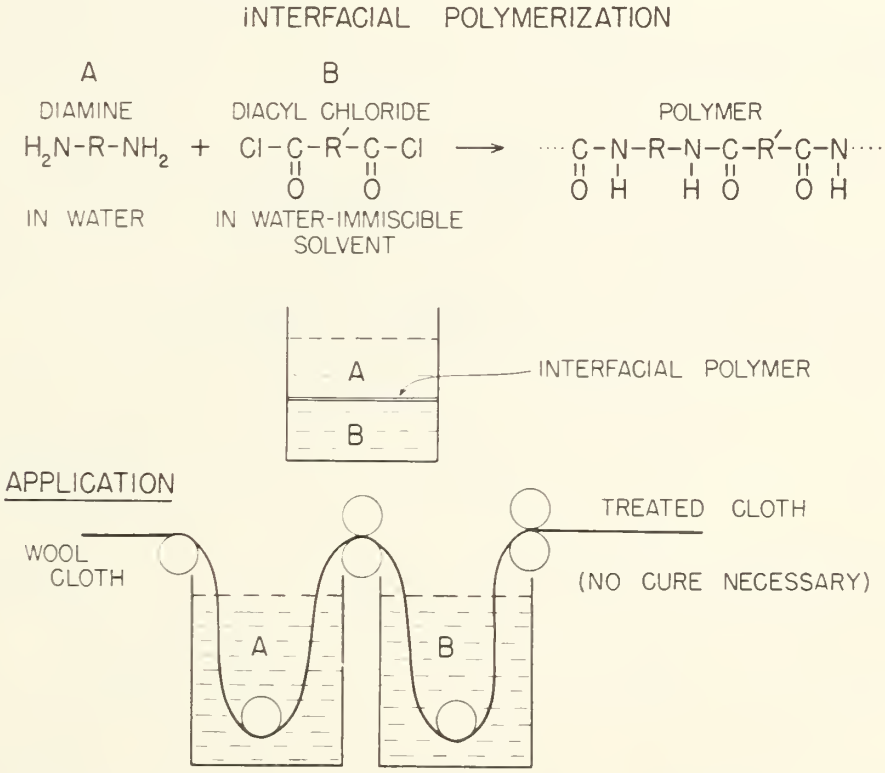

Figure 1. The principle of polyamide formation at an interface: "Interfacial Polymerization": (top) chemical formulas of reagents and product; (center) diagram of arrangement for forming polyamide at the boundary between immiscible solutions of the reagents in a beaker; (bottom) diagram of continuous application to fabric.

Application of resin to fabric by interfacial polymerization. The polymers formed have usually been made by other methods. IFP is of special interest because it is simple and versatile and is especially convenient and effective as a textile finishing treatment. Preformed polyamides, including polypeptides, and derivatives of polyamides have been applied to wool to effect shrink-resistance, but the use of IFP for this purpose is new. It has the advantage that adherent films of controlled thickness of a great variety of substances can be deposited quickly and continuously without the need of a special curing treatment.

In work summarized here, polyamides listed in Table 1 were deposited by IFP on wool flannel and various significant properties of the treated fabrics were evaluated. The cloth was degreased, rinsed, and conditioned before treatment. Typically, the conditioned, air-dry flannel was immersed for 15 seconds in a 4% aqueous solution of the amine. (It has been found advantageous to add sodium carbonate (e.g., 8% of the anhydrous salt) and 0.1% of a nonionic surfactant such as Triton X-100 to the amine solution.) The cloth was put through a clothes wringer to remove excess solution and then soaked for 15 seconds in a 4% solution of the acid chloride. The treated cloth was again put through the wringer, rinsed with water, and dried. Effects of many obvious experimental variations were studied to show their effects on the finished cloth. These variations included the solution concentrations, times of immersion, solvent used for the acid chloride, and order of application of reagents, in addition to different combinations of amines and acid chlorides.

Properties of experimentally treated fabric. Fabric samples given the various experimental treatments were screened for shrinkage protection by the two-minute Accelerotor test mentioned by Mr. Pardo. Wrinkle recovery, flexural rigidity, breaking strength, moisture regain, drying rate, soiling, color, and solubilities in acid, alkali, and after peracetic acid treatment were measured.

The best results so far have been obtained from hexamethylene diamine and aliphatic acid chlorides, especially sebacoyl (C_{10}) chloride. Effects of depositing various polyamides by IFP under comparable conditions is shown in Table 2. It is possible that some of these systems would give better results if tested under other conditions; but it should be noted that those giving the poorest protection were also those giving the weakest films by IFP in the absence of wool. Effects of experimental variables on the shrinkage protection afforded by the more favorable combinations are shown in Tables 3, 4, 5, and 6. Table 7 shows the degree of stability attained by some of these treatments toward repeated accelerated wash tests. The amount of polyamide deposited is shown in Tables 8 and 9 to be governed, at least in part, by the concentration of the reagents and their order of application, but not in this case by extraction of the flannel to remove the last traces of fatty material. In these tables, AM denotes the diamine component, AC the diacid chloride. The time of treatment is the time of immersion in each reagent solution; in Table 8, results are given separately for fabric treated without or with prior extraction with ether and alcohol, which resulted in minor differences in this instance. The resistance of the treated fabric to alkali, acid, and peracetic acid (oxidation) as determined by solubilization under standard conditions is not materially different from that of the untreated fabric.

Table 1. Polyamide systems studied.

Amine component	Acid chloride component
A. Both components aliphatic	
1,6-Hexamethylene diamine	Adipoyl chloride
"	Pimeloyl chloride
"	Azelaoyl chloride
"	Sebacoyl chloride
Piperazine	Adipoyl chloride
"	Sebacoyl chloride
B. Aliphatic amine, aromatic acid chloride	
1,6-Hexamethylene diamine	Terephthaloyl chloride
Piperazine	"
C. Aromatic amine, aliphatic acid chloride	
Metaxylylene diamine	Adipoyl chloride
"	Sebacoyl chloride
D. Both components aromatic	
Metaxylylene diamine	Terephthaloyl chloride

Table 2. Shrinkage protection obtained by interfacial formation of various polyamides under comparable conditions.

Polymer system	Resin deposited, %		Area shrinkage, % Accelerotor test of fabric treated	
	On fabric as received	On extracted fabric	as received	after extraction
None			52.0	
Hexamethylene diamine Sebacoyl chloride	3.4	2.4	5.0	7.9
Piperazine Sebacoyl chloride	4.5	2.7 to 4.4	32.8 to 45.2	32.8 to 45.2
Piperazine Terephthaloyl chloride	3.1	3.5	46.7	42.2
Metaxylylene diamine Sebacoyl chloride	6.0	3.2	21.7	31.2

Wool flannel was treated 20 seconds in 4% aqueous amine with 8% sodium carbonate (anhydrous) and 0.1% nonionic surfactant, then 20 seconds in 4% acid chloride in methylchloroform. Half of the tests, as indicated, were made with flannel that had been Soxhlet-extracted with ether and alcohol.

Table 3. Shrinkage protection obtained by interfacial formation of poly(hexamethylene-adipamide).

Reagents applied First	Second	Resin uptake, %	Area shrinkage, % Accelerotor, two minutes
None		None	52.0
18% AM	4% AC	7.5	2.0
4% AC	18% AM	4.3	3.0
9% AM	2% AC	4.7	13.6
4% AM	1% AC	2.4	10.7
1% AC	4% AM	1.6	17.2
9% AM	2% AC in toluene	5.5	3.0

Wool flannel was treated 15 seconds in each solution in the order given. Hexamethylene diamine (AM) was applied from water solution with 0.1% nonionic surfactant. Adipoyl chloride (AC) was applied from carbon tetrachloride except in the one instance noted.

Table 4. Shrinkage protection obtained by interfacial formation of poly(hexamethylene-pimelamide).

Reagents applied First	Second	Resin uptake, %	Area shrinkage, % Accelerotor, two minutes
None		None	49.0
4% AM	3% AC	1.4 to 2.0	2.0 to 4.9
3% AC	4% AM	1.0	1.0
2% AM	1-1/2% AC	less than 1	17.2
1-1/2% AC	2% AM	less than 1	5.9

Wool flannel was treated 15 seconds (60 seconds in the experiment of line 2) in each solution in the order given. Hexamethylene diamine (AM) was applied from water with twice as much sodium carbonate (anhydrous) as amine and with 0.1% nonionic surfactant. Pimeloyl chloride was applied from carbon tetrachloride.

Table 5. Shrinkage protection obtained by interfacial formation of poly(hexamethylene-azelaamide).

Reagents applied First	Second	Resin uptake, %	Area shrinkage, % Accelerotor, two minutes
None		None	52.0
4% AM	3% AC	1.5 to 2.7	2.0 to 3.0
3% AC	4% AM	1.0	6.9
4% AM	3% AC in Stoddard solvent	2.0	11.7

Wool flannel was treated 15 seconds (60 seconds in experiment of line 2) in each solution in the order given. Hexamethylene diamine (AM) was applied from water with twice its weight of sodium carbonate (anhydrous) and with 0.1% nonionic surfactant. Azelaoyl chloride was applied from carbon tetrachloride except as noted.

Table 6. Shrinkage protection obtained by interfacial formation of poly(hexamethylene-sebacamide).

Reagents applied First	Second	Immersion time, sec.	Resin uptake, %	Area shrinkage, % Accelerotor, two min.
None			None	52.0
AM	AC(CCl_4)	30	4.4	1.0
AM	AC(CCl_4)	15	4.6	4.0
AM	AC(CCl_4)	5	4.4	4.0
AC(CCl_4)	AM	90	3.2	2.0
AC(CCl_4)	AM	15	1.3	3.0
AM	AC(methyl-chloroform)	10	3.6	3.0
AM	AC(toluene)	30	5.9	0
AC(toluene)	AM	15	1.5	5.9
AM	AC(Skelly-solve B)	15	4.3	15.4
AC(Skellysolve B)	AM	15	2.2	3.0

Wool flannel was treated by immersion in each reagent for the time noted and in the order given. Hexamethylene diamine (AM) was applied as a 4% solution in water with 8% sodium carbonate (anhydrous) and 0.1% nonionic surfactant. Sebacoyl chloride was applied as a 2% solution in the solvent indicated.

Table 7. Durability of shrinkage protection obtained with polyamides.

Resin uptake, %	Area shrinkage, %				
	Number of two-minute Accelerotor washes				
	1	2	3	4	5
None	52				
Resin: Poly(hexamethylene-sebacamide)					
5.9	0	1	3	4	10
4.4	1	10			
3.2	2	10			
2.2	3	18			
Resin: Poly(hexamethylene-adipamide)					
7.5	2	7	16		
5.5	3	12			
4.3	3	15			

Table 8. Effect of reagent concentration on resin uptake.

Reagent applied		Polyamide uptake	
		On flannel	On solvent
First	Second	as received, %	extracted flannel, %
17.6% AM	4.0% AC	7.3	7.5
8.8% AM	2.0% AC	4.9	4.7
4.4% AM	1.0% AC	3.6	2.4
2.2% AM	0.5% AC	2.0	2.1
4.0% AC	17.6% AM	4.8	4.3
2.0% AC	8.8% AM	3.5	2.7
1.0% AC	4.4% AM	2.5	1.6
0.5% AC	2.2% AM	1.5	1.6

Wool flannel was immersed 15 seconds in each solution. Hexamethylene diamine (AM) was applied from water solution. Adipoyl chloride (AC) was applied from solution in carbon tetrachloride.

Table 9. Effects of time and order of treatment on resin uptake.

Reagent applied		Immersion time, seconds	Resin uptake, %
First	Second		
AM	AC	90	6.0, 8.3
AM	AC	30	4.4, 4.7
AM	AC	15	4.1, 4.6
AM	AC	5	4.2, 4.4
AC	AM	90	3.2, 3.5
AC	AM	15	1.3, 1.4

Poly(hexamethylene-sebacamide) was formed using 4% aqueous hexamethylene diamine (AM) and 3% sebacoyl chloride in carbon tetrachloride.

Table 10. Properties of worsted flannel treated by interfacial formation of poly(hexamethylene-sebacamide).

Polyamide on fabric, %	Area shrinkage % (Accelerotor, two min.)	Wrinkle recovery (warp)	Breaking load lbs.	Elongation at break %	Flexural rigidity mg.-cm.
None	52	146	36.4	33.4	143
1.4	3	149	35.2	35.2	-
3.5	2	146	39.0	36.0	296
4.1	4	146	40.1	34.6	210
4.7	1	149	38.0	32.0	160
6.0	3	149	37.6	33.9	194

The test samples were treated, rinsed, dried at room temperature, and the physical properties determined without prior mechanical conditioning.

Table 10 shows some of the physical properties of textile interest for flannels with various mounts of polyamide from hexamethylene diamine and sebacoyl chloride. Fabric samples with good shrinkage protection have been made repeatedly that do not differ appreciably from the original in rigidity, color, regain, and rate of drying. The treated fabrics soil slightly more with carbon black, slightly less with vacuum cleaner dirt than before treatment. Wrinkle-recovery and smoothness after tumble drying may be better than before treatment.

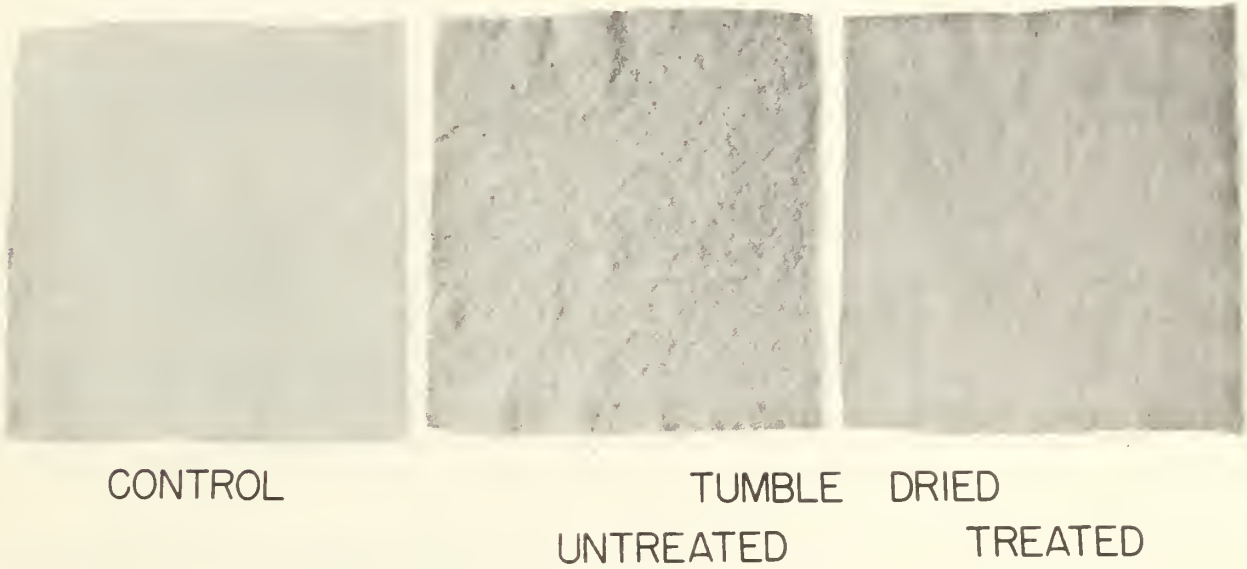

Figure 2. Wrinkle recovery of wool flannel finished with polyamide by interfacial polymerization. Control: Pressed fabric. Untreated and treated samples were wet out and tumble dried. The treated sample had a resin uptake of about 2 to 3% of poly(hexamethylene-sebacamide).

Conclusion. Durable films of polyamides, such as poly(hexamethylene-sebacamide), can be conveniently deposited by interfacial polymerization on the fiber surfaces of wool fabrics. Treated fabrics can be made that are dimensionally stable through accelerated wash tests and practically unchanged or improved in other properties.

Discussion of Dr. Whitfield's Report

Williams: How does fabric treated with preformed dissolved polyamide compare with fabric treated by interfacial polymerization?

Whitfield: Australian workers (see, for example, "Modification of Wool by the Application of Synthetic Polyamides," J. Delmenico, D. L. C. Jackson, and M. Lipson, Textile Research Jour. 24: 828, 1954) have studied the use of preformed polyamides (from hexamethylene diamine and adipic acid) and derivatives. Fairly good results were obtained by depositing water-soluble derivatives and then removing the groups conferring water solubility. This process has not proved commercially practical. We have not made an extensive comparison, but have so far been unable to deposit an effective coating of the preformed polymer.

Williams: Does the treatment have a moth-proofing effect?

Whitfield: This aspect has not been studied. It seems unlikely that the polyamide coating will of itself give protection. However, it is possible that suitable ingredients may be incorporated in one or the other of the treating solutions.

Question: What is the mechanism of the shrink-resisting action of the IFP treatment?

Whitfield: We have little evidence bearing on this subject. Measurement of the differential frictional effect (difference in the frictional coefficients in the two directions) of single fibers removed from a treated flannel that had complete protection against shrinkage showed only a slight decrease, scarcely enough to account for the protection. Extraction of this fabric with a good solvent for polyamide did not remove the polymer. We originally supposed that such a treatment would remove deposits of resin large enough to act as "spot welds" between fibers. The basis of the protection evidently needs further study.

Question: Is there a reaction between the acid chloride and wool itself?

Whitfield: Because of experiments like that just mentioned, in which polyamide cannot be extracted from treated fabric with good solvents, we believe that the polyamide is grafted to the wool. This can be accounted for by reaction between wool and acid chloride during the treatment. Certainly one would expect free amino and hydroxyl groups on the wool surface to react with acid chloride. However, reaction of wool with acid chloride alone has given no shrinkage protection and very little increase in weight.

POLYAMIDE SHRINK-RESIST FINISHES FOR WOOL BY INTERFACIAL POLYMERIZATION
II. PILOT SCALE STUDIES OF PROCESSING VARIABLES

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This report summarizes pilot-scale study of interfacial polymerization (IFP) to develop a practical continuous process for treating wool fabrics. Effects of important process variables have been defined by statistically planned experiments. Second, representative woolen and worsted fabrics, knitted goods, and blanket material have been treated and evaluated. Finally, finishing procedures needed after the treatment for satisfactory results have been determined. Suggestions for practical trial, based on our experience to date, are given in a Correspondence Aid 74-9, obtainable on request.

Choice of reagents and solvent. Of the many combinations of reagents possible for applying a polyamide to wool by IFP, hexamethylene diamine (HMDA) and sebacoyl chloride were chosen for detailed study. This pair is among the most effective. More important, both reagents are potentially readily available, although neither is now sold in large quantities. HMDA is used by the ton in making nylon fiber and molding powder; almost its entire production is captive. Sebacoyl chloride is made from sebacic acid, available in commercial amounts from castor oil. These compounds are currently available as chemical specialties at about $2 per pound. Other, potentially cheaper acid chlorides have been compared.

Although a chlorinated solvent for the acid chloride gives better shrink-resistance, its volatility, toxicity, and cost militate against its practical use. Therefore the possible use of cheaper, less toxic petroleum solvents with relatively high flash points, such as Stoddard solvent, has been emphasized.

Methods of application. This report concerns the development of the dip-and-pad technique, in which the polyamide resin is applied by running the fabric through the two reagent solutions successively, using squeeze rolls after each dip to remove excess solution. Other methods have been explored with some success. For example, sweaters and socks have been treated by the batch, removing excess solution after each dip by centrifuging. (This process was demonstrated in the Processing Laboratory Thursday afternoon.) It is also possible to apply the solutions by spraying and to remove the excess by vacuum extraction, but these methods have not been studied in detail.

Preliminary development of the dip-and-pad method. Continuous dip-and-pad application was first tested with narrow fabric using a small laboratory padder for both steps. With this padder, serious deposition of excess resin occurred on the pad rolls and on the fabric unless (a) the speed of processing was extremely low, about 1/2 yard per minute, (b) the padding pressure was as high as possible, about 80 pounds per linear inch, (c) the concentration of acid chloride was at least 3%, and (d) carbon tetrachloride was used as solvent

for the acid chloride. Under otherwise optimum conditions, use of Stoddard solvent in place of carbon tetrachloride was completely unsatisfactory because of excessive, irregular surface resin deposition.

Avoiding excess surface deposition. These undesirable surface deposits result from intermixing excess aqueous and organic treating solutions when conditions are such that they are expressed together in the second padding step. They are less likely to occur if the aqueous diamine solution is applied first, followed by the nonaqueous acid chloride. At least two other practical considerations make it reasonable to limit development to this normal order of treatment. First, the pickup of organic solvent is much less on the amine-saturated, wet fabric than on the dry fabric, so that the solvent requirement is much lower with the normal order. In addition, when the acid chloride is applied from a petroleum solvent as the second step, aqueous amine solution that may get into the second bath from the second squeeze rolls settles, because of its greater density, so that it can be prevented from contaminating the fabric during the solvent dipping step. The most important requirement, however, is that sufficient amine solution be removed from the fabric by the first padding step that further expression of this phase is negligible during the second padding.

The next development was to use a full width 10-ton production textile padder equipped with a variable speed drive and controls for adjusting the padding pressure to more than 400 pounds per linear inch. Specially constructed immersion pans permitted dipping and padding narrow width fabrics through one solution on the left half of the pad, then through the other on the right half with minimum delay. (This narrow width experimental treating procedure was shown in the Processing Laboratory on Thursday afternoon. Samples of the treated cloth were distributed the next morning.)

With this equipment, deposition of excess surface polymer was entirely avoided by working at the lowest possible speed, 2 yards per minute, and maximum pressure during the amine padding, with somewhat lower pressure during acid chloride padding. Equally important, the higher padding pressure gave a very significant improvement in shrinkage control. This improvement appreciably decreased the resin uptake needed and permitted use of Stoddard solvent. The lower resin requirement, besides lowering the chemical cost, minimized changes in the hand of the treated fabric and in whitening when IFP is applied to dyed material.

Significant processing variables. Processing variables suspected of affecting the results were next examined systematically in a series of tests planned with the help of our staff statistician, Miss Marion Sandomire. Only one fabric was used, a stock-dyed worsted flannel, very high shrinking and among the more difficult to stabilize dimensionally. The bolt was cut into pieces 14" by 60" to test the various conditions of treatment. The pertinent variables are listed in Table 1. For each of the seven factors studied, the level of treatment given first is known from prior experience to control shrinkage of the standard fabric.

The first variable is the solvent used for the acid chloride. Stoddard solvent, used extensively for dry cleaning, has been taken as standard. It is an inexpensive, aliphatic hydrocarbon solvent with a relatively high flash

Table 1. List of processing variables tested.

I. Solvent:
(1) Stoddard, essentially 100% aliphatic petroleum solvent with high flash point
(2) Socal 25, essentially 100% aromatic petroleum solvent with high flash point
(3) Chlorothene (methyl chloroform), low toxicity chlorinated solvent

II. Padding speed (same for aqueous and solvent pass):
(1) Low speed: 2 YPM
(2) High speed: 12 YPM

III. Concentration ratio (weight percent diamine in aqueous phase to weight percent acid chloride in solvent phase):
(1) 2/3; (2) 2/2; (3) 1/3; (4) 2/1; (5) 3/1

IV. Padding pressure (during solvent pass):
(1) 40 psig.
(2) 60 psig.

V. Sodium carbonate concentration (in aqueous phase):
(1) Twice diamine conc.
(2) One-half diamine conc.

VI. Wetting Agent (in aqueous phase):
(1) Nonionic surfactant
(2) Phosphate ester of nonionic surfactant

VII. Acid:
(1) Sebacoyl chloride, C_{10} diacid chloride
(2) Azelaoyl chloride, C_9 diacid chloride
(3) Adipoyl chloride, C_6 diacid chloride

Table 2. Summary of variables studied in the four separate factorial experimental series.

Experimental plan:	I	II	III	IV
Variable	Number of variations studied			
Solvent	3	1	1	1
Speed	2	2	2	2
Concentration ratio	5	2	2	5
Pressure	1	2	1	1
Sodium carbonate	1	2	1	1
Wetting agent	1	1	2	1
Acid chloride	1	1	1	3

point. Socal 25 is a comparable aromatic hydrocarbon solvent. Chlorothene (methylchloroform) is a relatively little toxic chlorinated solvent.

Since it is important to use the highest practical processing speed, 12 yards per minute, the highest attainable with the 10-ton padder, was compared with the slow 2 yards per minute found adequate to give good control.

Use of 2% diamine (by weight) and 3% acid chloride in the treating solutions can control shrinkage of the standard flannel. Other combinations of concentrations, conveniently expressed as the ratio of weight percentage of the amine solution to that of the acid chloride solution, were tested. These were selected to show possible decreases in chemical costs as well as trends in shrinkage protection.

The highest possible pressure, 60 pounds per square inch, gauge, was always used for padding the aqueous amine. The lower standard pressure, 40 psig, for the solvent pass was compared with a higher pressure, again 60 psig.

Sodium carbonate is added to the aqueous amine solution to neutralize the acid formed by the reaction. The concentration normally used (calculated in terms of the anhydrous salt) is twice that of the hexamethylene diamine. This amount is approximately equivalent to the acid released by complete reaction, assuming a final pH of 8 to 9. A carbonate concentration of one-half the amine concentration was chosen for comparison, since this would lower the requirement for acid for subsequent neutralization and would improve wetting action of the amine solution, especially at the higher diamine concentrations.

A phosphate ester of a nonionic surfactant was compared with the alkylaryl polyether alcohol normally used as a wetting agent in the aqueous diamine solution. The phosphate ester is reported by the manufacturer to be particularly effective at high alkalinity.

Azelaoyl chloride and adipoyl chloride were compared with the sebacoyl chloride chosen as the standard diacid chloride reagent. These chlorides are derived from the unbranched, saturated dicarboxylic acids with respectively 9, 6, and 10 carbon atoms. The former were studied because they are potentially cheaper than sebacoyl chloride.

Hexamethylene diamine was used in all treatments.

To limit the tests to a workable number, 66, reasonable assumptions were made that certain of these factors do not interact, allowing certain combinations to be disregarded. Four separate factorial plans resulted, shown in Table 2. The major plan, I, compares the three solvents, five reagent concentration ratios, and two processing speeds. Plan II defines effects of pressure in the second padding step and sodium carbonate concentration. Plan III shows the effect of choice of wetting agent. Plan IV shows the effect of choice of acid chloride.

Initial interpretation of results is based on resin uptake and total felting shrinkage resulting from two 75-minute accelerated laundering tests.

Solvent type used for the acid chloride. Figure 1 summarizes the results of Plan I. Fabric shrinkage is plotted against resin uptake, with which it is usually directly correlated. Results for the two processing speeds are separately shown and the five reagent concentration ratios are identified.

The best shrinkage protection results from use of the chlorinated solvent, next best from Socal 25, and the poorest from Stoddard solvent. Nevertheless use of Stoddard solvent may be preferred for the following reasons. The high cost of a chlorinated solvent would add to the cost of treatment and would probably require a solvent recovery system. On the other hand, Socal 25 has the disadvantages of higher toxicity due to its aromatic composition, a stronger odor that may be objectionable in mill operations, and greater difficulty of removal from the fabric after treatment.

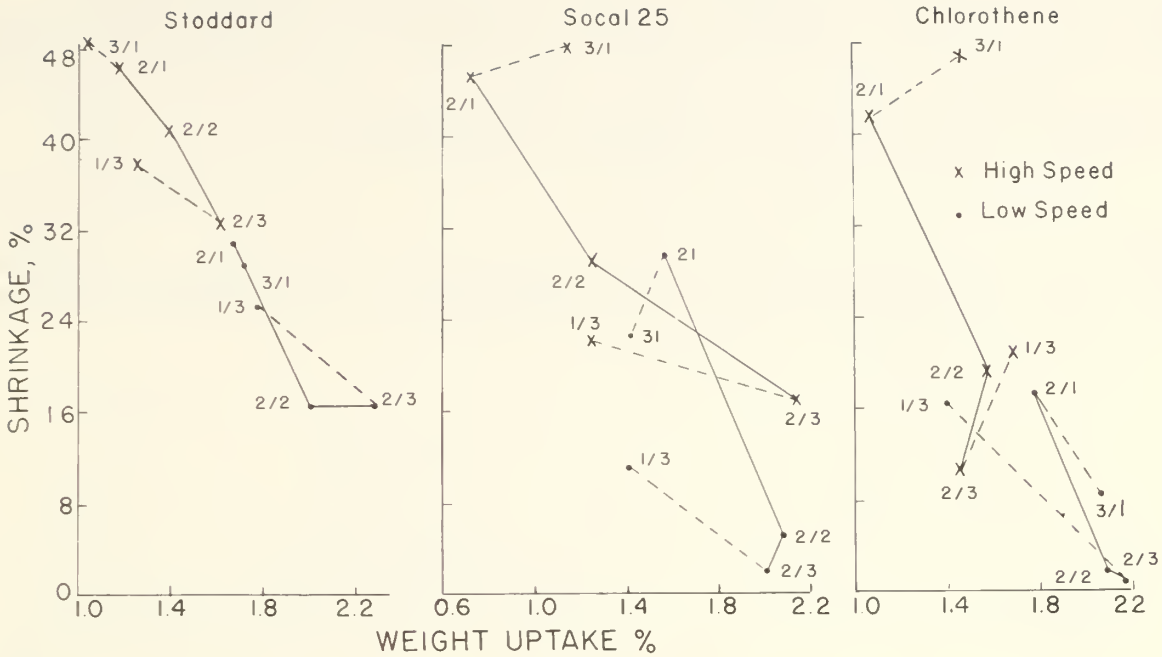

Figure 1. Effects of choice of solvent, processing speed, and ratio of concentration of amine to acid chloride on shrinkage protection obtained with interfacially formed poly(hexamethylene-sebacamide).

In spite of the distinct effect of solvent type on shrinkage control, it is specially noteworthy that the solvent type does not have a significant effect on the resin uptake, other factors being the same. The better shrinkage control resulting from use of chlorothene or Socal 25 must therefore be sought in properties such as the molecular weight of the resin formed or the uniformity or adherence of the deposited film.

Processing speed. Processing at 12 yards per minute rather than 2 yards per minute results in distinctly lower shrinkage protection. On the average, the resin uptake is 25% less at the higher speed, and the shrinkage about twice. This influence of processing speed shows that the time needed for IFP is critical. Since the chemical reaction is reported to be extremely fast, it seems likely that the rate controlling process is diffusion of the reagents to the site of reaction. Under the conditions tested, about 50 seconds elapse at the lower speed between immersion of the diamine-saturated fabric in the acid chloride solution and passage through the squeeze rolls. About 8 seconds are allowed at the higher speed. It is probable that appropriate design of the dip pans and of the fabric threading will permit relatively longer dip time and longer delay between dipping and padding, making feasible processing at higher speeds.

Reagent concentration and concentration ratio. The results of Plan I also show the relationships of shrinkage protection and reagent concentration. When 2% diamine solution is used with the high processing speed, resin uptake

and protection increase regularly as the acid chloride concentration is increased from 1 to 3%. However, at the slower speed for which the contact time is not limiting, increase in the acid chloride concentration above 2% has no further effect. Work is planned to show whether this limiting concentration of acid chloride depends on the diamine concentration, especially with fabrics that can be stabilized with low diamine concentrations.

Increasing the diamine concentration from 2 to 3% increases the resin uptake and protection if sufficient acid chloride is available. If the acid chloride is low enough to be limiting (1%), increasing the diamine concentration from 2 to 3% actually decreases the shrinkage control. This observation emphasizes the importance of the acid chloride concentration.

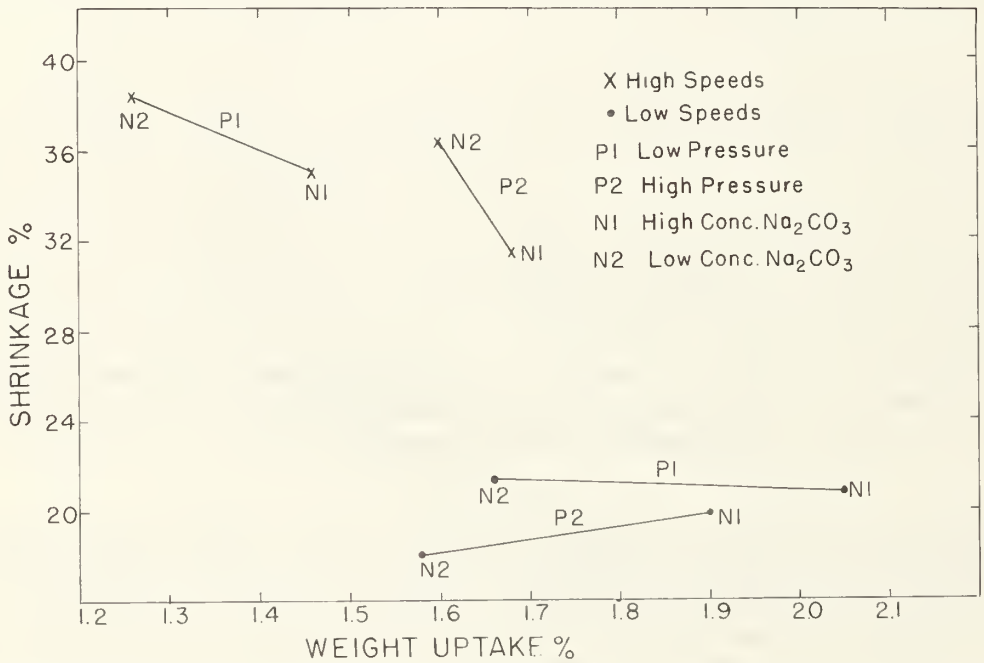

Figure 2. Effects of processing speed, pad roll pressure, and sodium carbonate concentration on polyamide uptake and shrinkage protection.

Padding pressure after the acid chloride dip. Figure 2 summarizes results of Plan II. Two padding pressures were used following acid chloride treatment; two sodium carbonate concentrations were used in the diamine solution; and two ratios of diamine to acid chloride. Shrinkages observed are plotted against resin uptake. Results for the two concentration ratios are consistent, so that averages are shown. Shrinkage control is slightly, but significantly, better with use of higher padding pressure. This corresponds to the improvement resulting from using the full-size padder instead of the laboratory model. The improvement resulting from heavier padding pressure may be due to increased physical bonding of fibers and yarns brought into closer contact. In spite of this improvement, somewhat lower padding pressure should be used in the acid chloride padding step to minimize squeeze-out of aqueous amine solution during this second padding.

Sodium carbonate concentration in the aqueous diamine solution. The lower sodium carbonate concentration is always associated with lower resin uptake. However, at the lower speed, shrinkage protection is not affected.

Wetting agent used in the aqueous diamine solution. The tests of Plan III show that better shrinkage protection was obtained with the nonionic alkaryl polyether alcohol than with the phosphate ester wetting agent chosen for comparison.

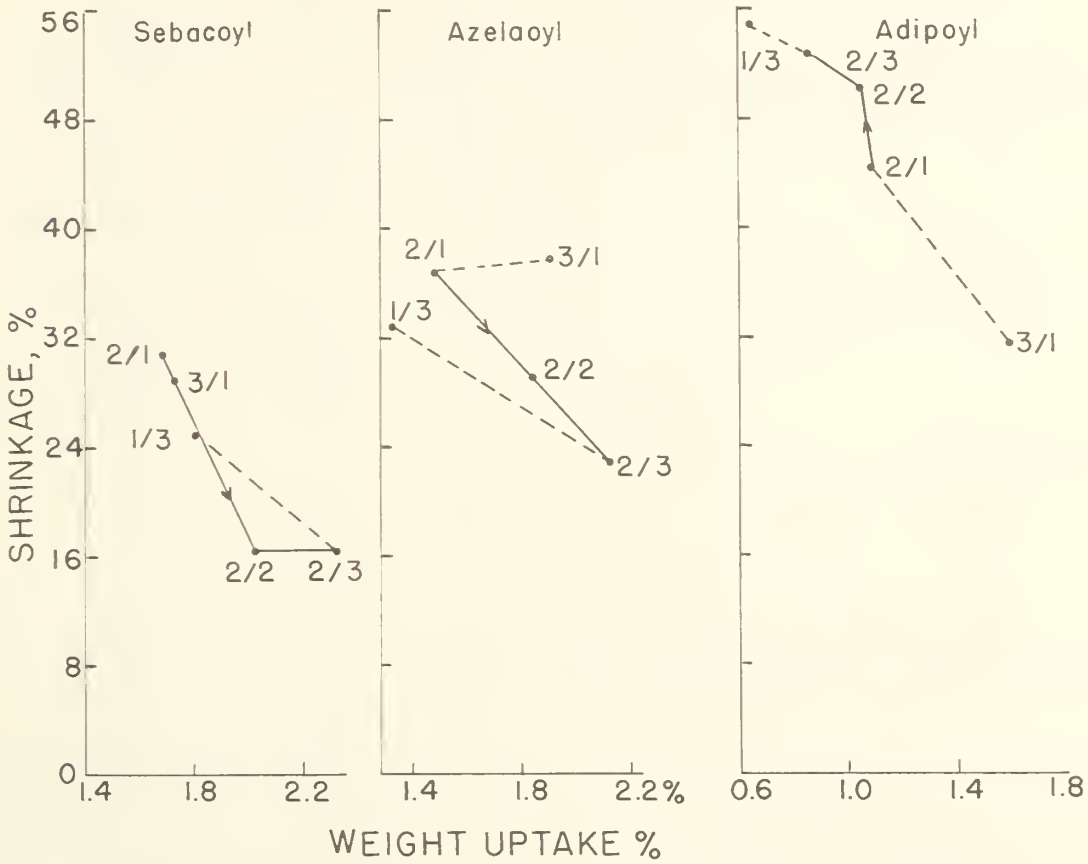

Figure 3. The effect of choice of acid chloride and the concentration ratio of amine to acid chloride on polyamide uptake and shrinkage protection.

Selection of acid chloride. Figure 3 compares shrinkage *versus* resin uptake for the acid chlorides of sebacic, azelaic, and adipic acids (with 10, 9, and 6 carbon atoms, respectively) applied from Stoddard solvent. Sebacoyl chloride is seen to be the most effective and azelaoyl next, while adipoyl is ineffective under the given experimental conditions. Weight uptakes with the sebacoyl and azelaoyl chlorides are about equal, while those with adipoyl chloride are much lower. Since azelaoyl and adipoyl chlorides are less expensive, these results are disappointing and suggest further exploration for effective processing conditions. The consistent increase of effectiveness with molecular weight suggests that even better results might be obtained with longer chain diacid chlorides.

Table 3. Application of IFP treatment to various woolen fabrics. System: hexamethylene diamine (HMDA) followed by sebacoyl chloride in Socal 25.

Fabric	HMDA,%	Weight uptake,%	Area shrinkage,%[1/]
Flannel, piece-dyed	Control	-	32.2(1)
	1/4	0.17	29.0
	1/2	0.61	14.8
	1	1.15	3.0
	2	1.70	2.2
Flannel, 85% wool 15% nylon, undyed	Control	-	24.3(1)
	1/4	0.59	4.4
	1/2	0.83	2.8
	1	1.39	2.2
	2	2.08	0
Plaid shirting	Control	-	36.0(1)
	1/4	0.13	23.6
	1/2	0.27	15.6
	1	0.98	9.0
	2	1.45	+0.4
Napped coating	Control	-	31.8(1)
	1/4	1.18	10.6
	1/2	1.38	5.2
	1	2.18	1.6
	2	2.59	+0.3
Military shirting, wool-nylon blend	Control	-	22.4(1)
	1/4	0.41	22.0
	1/2	0.60	14.5
	1	1.37	7.1
	2	1.72	5.8

1/ After 4 75-min. accelerated wash test cycles, except controls 1 cycle.

Response of representative fabrics to IFP. Polyamide finishes have been applied by IFP to a great variety of fabrics. These include, in addition to the several types used in developing the treatment, materials submitted by a wide sector of the woolen, worsted, and felting industry. In screening the fabrics to determine the required treatment level, the material supplied is first scoured in a dye beck with nonionic surfactant to remove residual processing oils, softeners, and other chemical agents that may remain from previous processing. This prescour also serves to reduce residual relaxation shrinkage and to indicate the wash-fastness of dyed fabrics.

Woolens. Table 3 summarizes typical results for various woolen fabrics. These data were obtained by treating narrow widths in hexamethylene diamine solutions of 1/4 to 2%, as shown in the first column, followed by treatment

Table 4. Application of IFP treatment to various worsted fabrics. System: hexamethylene diamine (HMDA) followed by sebacoyl chloride in Socal 25.

Fabric	HMDA,%	Weight uptake,%	Area shrinkage,%[1/]
Sheen gabardine suiting	Control	-	15.1(1)
	1	0.52	24.3
	2	1.35	11.7
	3	1.75	10.2
Flannel suiting	Control	-	24.9(1)
	1	1.20	24.0
	2	1.61	7.9
	3	1.94	5.6
Worsted suiting	Control	-	28.7(1)
	1/2	1.16	30.0
	1	1.50	17.4
	2	2.21	6.6
	3	2.57	4.7
Uniform fabric	Control	-	24.8(1)
	1/2	0.73	32.9
	1	1.19	22.7
	2	1.91	16.1
	3	2.26	13.6

1/ After 4 75-min. accelerated wash test cycles, except controls 1 cycle.

with 3% sebacoyl chloride in Socal 25. There is a fair correlation between the concentration of HMDA and weight uptake, which depends also on the wet pickup after padding. Area shrinkages shown in the last column resulted from four 75-minute accelerated wash test cycles in a home-type washer, except for values for untreated fabric, which were obtained after a single 75-minute wash. Full width treatment of the fabrics was then accomplished successfully using the optimum diamine concentration indicated by the narrow width screening tests.

In spite of the fairly high shrinkage of the untreated fabric, woolens as a class are relatively easy to stabilize by IFP. Treatment under optimum conditions of speed, pressure, solvent, and acid chloride concentration rarely requires a diamine concentration above 1%. In many instances, as shown for a wool-nylon flannel, as little as 1/4% hexamethylene diamine gives excellent results.

Worsteds. Table 4 illustrates shrinkage protection of worsted fabrics obtained by IFP. In spite of the comparatively low shrinkage of the untreated material, worsted fabrics as a class are relatively more difficult to stabilize. Most worsteds have been found to require at least a 2% concentration of HMDA for effective control. However, the wet pickup of worsteds is usually at least 20% less than for woolens, so that for equal diamine concentrations, the

Table 5. Application of IFP treatment to knit goods and blanket. System: hexamethylene diamine (HMDA) followed by sebacoyl chloride in Socal 25.

Fabric	HMDA,%	Weight uptake,%	Area shrinkage,%[1/]
Jersey, undyed	Control	-	51.2(1)
	1/2	0.53	39.6
	1	1.25	15.3
	2	1.82	2.2
	3	2.55	1.6
Jersey, dyed	Control	-	57.6(1)
	1	1.29	18.0
	2	2.56	0.7
	3	3.04	0.6
Blanket, dyed, unnapped	Control	-	58.3(1)
	1/4	0.39	53.8
	1/2	0.53	42.1
	1	1.54	20.0
	2	1.92	3.2

1/ After 4 75-min. accelerated wash test cycles, except controls 1 cycle.

actual amount taken up by the fabric is materially less for the worsted.

The greater difficulty of stabilizing worsteds is probably due to the high twist of the yarn and to the compact weave which hinder penetration of the treating solutions, especially the organic acid chloride solution. In addition, latent relaxation shrinkage, particularly in the warp direction, contributes to the difficulty of shrinkage control. For instance, most of the shrinkage of the sheen gabardine and uniform fabrics treated with 2 and 3% HMDA, is in the warp direction. Since the fill shrinkage is controlled, the warp shrinkage may be attributed to relaxation or consolidation rather than felting.

Knit goods and blanket material. Table 5 shows results of IFP on knit goods and blanket material. The exceptional tendency of the untreated materials to shrink is shown by the result of a single 75-minute wash. The efficacy of IFP treatment in these cases is correspondingly impressive: essentially complete control of shrinkage is achieved with a HMDA concentration of 2%. In treating the jersey, the material was processed in tubular form. Except for a somewhat high squeeze-out of amine solution during acid chloride padding, treatment presented no special problems. Treatment of tubular knit goods by padding seems quite feasible. In the treatment of blankets, too, excess squeeze-out of amine solution occurred in the acid chloride padding step because of the thick, bulky structure; otherwise processing was normal.

Finishing after IFP. Fabric treated by IFP emerges from the second padding step somewhat stiff and boardy, saturated with solvent, and

contaminated with salts and unreacted reagents. Since the resin is essentially cured practically as soon as it is formed in the second dipping and padding step, the material may be scoured directly, without intermediate drying or curing. Scouring is exceedingly important, not only to remove solvent and other contaminants, but more particularly because the mechanical working restores to a high degree the hand and suppleness characteristic of the original materials.

In the treatments described, it has been found that 20 to 30 passes through the squeeze-rolls of a dolly washer are completely adequate to remove the solvent and to restore the flexural rigidity of the material to approximately that of the original. In this scour, 0.1% of a nonionic wetting agent, with acid equal to 1% of the fabric weight, has given good results. The acid is needed to neutralize excess sodium carbonate, which would otherwise make the finished fabric somewhat harsh. The fabric is rinsed after scouring. Addition of 0.1 to 0.2% of a textile softening agent (based on the weight of the fabric) at this stage greatly improves the hand.

After washing, the fabric is pin-tenter dried at a width generally set one inch narrower than the wet width of the fabric. In addition, the dryer is overfed to reduce residual warp relaxation shrinkage. Normal drying temperatures and speeds suffice to remove traces of solvent that may remain.

Although the treated fabrics respond normally to pressing and decating, final dry finishing steps will depend on the fabric and the desired hand.

Dyeing. Dyeing of fabric treated by IFP has been studied with only two classes of wool dye: acid premetallized and acid milling. With each, dyeing is normal except that the rate of exhaustion is somewhat faster and the shade of IFP treated material somewhat deeper than that of an untreated piece dyed at the same time. Dyeing was exceptionally level, an indication of the uniformity of the treatment. For two fabrics and two dyes studied, after-dyeing has little or no effect on the shrinkage control.

When polyamide is applied by IFP to dyed material, experience shows that if the dye is wash-fast under alkaline conditions, little or no bleeding occurs in the diamine padding step. Some materials received that were not wash-fast bled profusely in the diamine solution.

Dyed material tends to be slightly whitened by IFP. The effect is very slight below 1% weight uptake. At higher levels it may be necessary to change the dye formula to compensate for the shade change.

Napping. Effects of IFP on the napping of blanket material and of napping on shrinkage control have been examined with cooperation of interested manufacturers. Limited napping trials show treated material somewhat harder to process than untreated material of comparable quality. The effect of napping on shrinkage control was explored by comparing shrinkages of IFP treated material unnapped, napped, and napped after dyeing. The sample napped without dyeing shrank slightly more than the unnapped sample, but the difference was not marked until the third 75-minute accelerated wash test. The sample napped after dyeing shrank somewhat more. This additional shrinkage is attributed to 10% stretch in the fill direction which occurred during processing.

Summary. The main difficulty in producing shrink-resistant wool fabrics by IFP is the need for using an organic solvent. However, in the normal treatment, the solvent uptake is less than 20% of the fabric weight. The potential fire hazard in using a hydrocarbon solvent can be decreased by removing the solvent by scouring rather than drying. Other requirements are adequate ventilation and care in handling the diamine and acid chloride treating solutions.

On the other hand, IFP is readily adapted to control shrinkage of a wide range of woolen and worsted fabrics, woven and knitted. The shrinkage control attainable with very low resin uptake is outstanding. Effects on chemical and physical properties are minor. The hand of treated material is, in many cases, nearly indistinguishable from that of the original fabric. Treated fabric is unusually wrinkle-free after washing and tumble-drying.

The treatment can be carried out continuously with conventional textile processing equipment. Fabric hand is not critically affected by overtreatment, but control of the process is relatively simple.

All present experience indicates that shrinkage control of wool fabrics by IFP is technically feasible. Factors that deserve further consideration before proceeding to plant trials are: (1) means of attaining optimum processing speeds; (2) use of solvent of higher flash point; (3) possible need for controlling diamine and acid chloride concentrations in an extended run; and (4) further adjustment of processing variables to reduce costs.

Discussion of Mr. Fong's Report

Kennedy: Do you have photomicrographs showing the degree of coverage of fibers from resin-treated fabrics?

Lundgren: This sort of work is just beginning. It appears that the film is very thin, although not extractable by solvents for polyamides. Epoxy films have been demonstrated in some cases by selective staining. We hope to do more of this.

Kennedy: Can IFP be applied to top? What effect does this have on spinning?

Fong: In our experience, IFP treatment of top prevents drafting.

Kennedy: What happens to the tear strength of IFP treated fabrics?

Fong: The tear strength is usually slightly decreased - about 5 to 10%.

Kennedy: Do IFP treated fabrics pill after laundering?

Fong: We haven't had the Harris laboratory test this yet. Our experience suggests that pilling is not a problem.

Kennedy: Is there any difficulty from unwanted wrinkling resulting from IFP?

Fong: No.

Kennedy: Can you say anything further about the effect of the treatment on dyeing?

Fong: We have tested very few examples. Experience has been limited to the acid milling and premetallized dyes, as described.

Kennedy: What effect does IFP have on the abrasion resistance of fabric?

Fong: In one trial with the Stoll Flex-abrader, where the untreated fabric failed after 360 cycles, the treated material lasted until 450. This result may be misleading because the treated sample had also been finished with a softening agent.

Question: Is it possible to remove the polyamide finish?

Fong: No.

Question: What is the mechanism by which IFP controls shrinkage?

Whitfield: This hasn't been thoroughly studied. The fact that thorough extraction of treated fabric with good solvents for the polyamide does not affect the shrinkage protection suggests that interfiber bonding or "spot welding" may not be a sufficient explanation.

Dusenbury: It would be informative to try experiments like those of Royer and McCleary of the American Cyanamid Company, who looked for changes in the torsional modulus of single fibers as a result of stretching enough to break a surface film.

Laxer: Does the IFP treatment have much effect on knot strength?

Fong: Knot strength is not much changed.

Laxer: Is IFP a satisfactory blanket treatment?

Fong: I don't think we have enough information to answer this question definitely.

Poats: Do you believe that the ten-carbon-atom chain length for the acid chloride is optimum?

Fong: We plan to study other, especially higher, members of the series to settle this question.

Question: I wonder whether you have studied reagents with odd numbers of carbon atoms in case these have any special significance?

Whitfield: We haven't studied any except pimeloyl and azelaoyl chlorides. Although physical properties of polyamides are reported to fluctuate somewhat according to oddness and evenness of the carbon chain length of the monomers, no effect of this sort on shrinkage control has so far been observed.

Comment: Although carbon tetrachloride seems to be the most effective solvent for IFP, I am concerned about its toxicity.

Fong: For this reason and because of cost carbon tetrachloride is not our preferred solvent. The aliphatic hydrocarbon solvent is cheap enough that it may not require solvent recovery measures. The fire or explosion hazard can probably be controlled by using a fraction with sufficiently high flash point.

Question: Does IFP affect the inflammability of the fabric? It occurs to me that the treatment may lead to a self-supporting char tending to increase inflammability.

Fong: We have no information on this subject.

Question: Is the treatment light fast?

Fong: Again we have no information.

Laxer: Now that felting shrinkage is somewhat under control, can consolidation shrinkage be avoided? What is your experience?

Fong: It is very difficult to stabilize against consolidation, which occurs gradually in the warp direction. Consolidation is retarded, but not eliminated by IFP. The problem may be one of fabric design.

Question: What about accumulation of aqueous amine solution in the acid chloride treating bath? Are special precautions needed to avoid irregular secondary surface deposits?

Fong: We do use one special arrangement. The fabric leaving the acid chloride dip is passed over a bar somewhat higher than the nip of the padding rolls, so that the material enters the nip at an angle from above. This keeps solution squeezed out from running back down the fabric. We haven't found it necessary to make any special provision for amine solution draining back into the acid chloride tank. In an extended run this may be desirable.

Question: Is it necessary to scour the fabric before IFP?

Fong: Prescouring is not always necessary. However, it permits the fabric to relax and gives a chance to correct excess acidity that may remain from dyeing and to remove processing oils or finishing agents that may be incompatible.

Question: Does hydrochloric acid accumulate as a result of reaction of the acid chloride?

Fong: Sodium carbonate is added to the amine bath to avoid this.

Question: Is there any serious loss of acid chloride in the second bath as a result of hydrolysis due to the amine solution carried over?

Fong: Apparently not.

Laxer: Do you think IFP will be applicable in the near future to woolens and knit goods?

Fong: We believe that this treatment is especially promising for woolens, but that it can also be used successfully with worsteds.

Laxer: Is it possible to apply durable creases after IFP treatment?

Lundgren: Yes. Durable creasing with ethanolamine is perfectly satisfactory after IFP.

Wolff: . . . I wish you would enlarge on the expression "graft polymer" that has been used several times in these reports.

Whitfield: Proteins such as wool keratin have many chemically reactive atomic groups. Typical examples are hydroxyl and amino groups. I mention these in particular because they are theoretically able to combine with acid chlorides and presumably will do so to the extent that they are accessible. In this way reactive sites of wool may serve as points of attachment for polyamide chains formed during IFP. This sort of process in which polymer of one sort is joined by durable chemical bonds to polymer of another sort (in this case wool keratin) has been described as grafting. The combination is called a graft polymer.

We haven't proved that polyamide is truly grafted to wool by IFP, but the relative durability of the treatment and insolubility of the applied film suggest that it may be grafted.

NEW CHEMICAL MODIFICATIONS OF WOOL

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(Dr. Wasley spoke instead of Dr. N. H. Koenig, who was unable to be present.) This report includes research on prevention of yellowing and chemical attack, as well as work on chemical compacting of felt and on durable creasing.

Prevention of yellowing due to light. In addition to studies of basic aspects of yellowing, which are discussed by H. P. Lundgren in Jour. Textile Institute, 51 (12, part 1): T806, Dec. 1960, screening tests of a great variety of substances have been carried out to determine possible activity in preventing yellowing by ultraviolet light. Special attention has been given to finding effective substances that can be applied from aqueous solution (Rose, Walden, and Moore, "Comparison of Ultraviolet Absorbers for Protection of Wool Against Yellowing," accepted by Textile Res. Jour.). An example of a sulfonated hydroxybenzophenone that has been found particularly effective is shown in Figure 1. This substance, at an uptake of about 8 percent on flannel, had

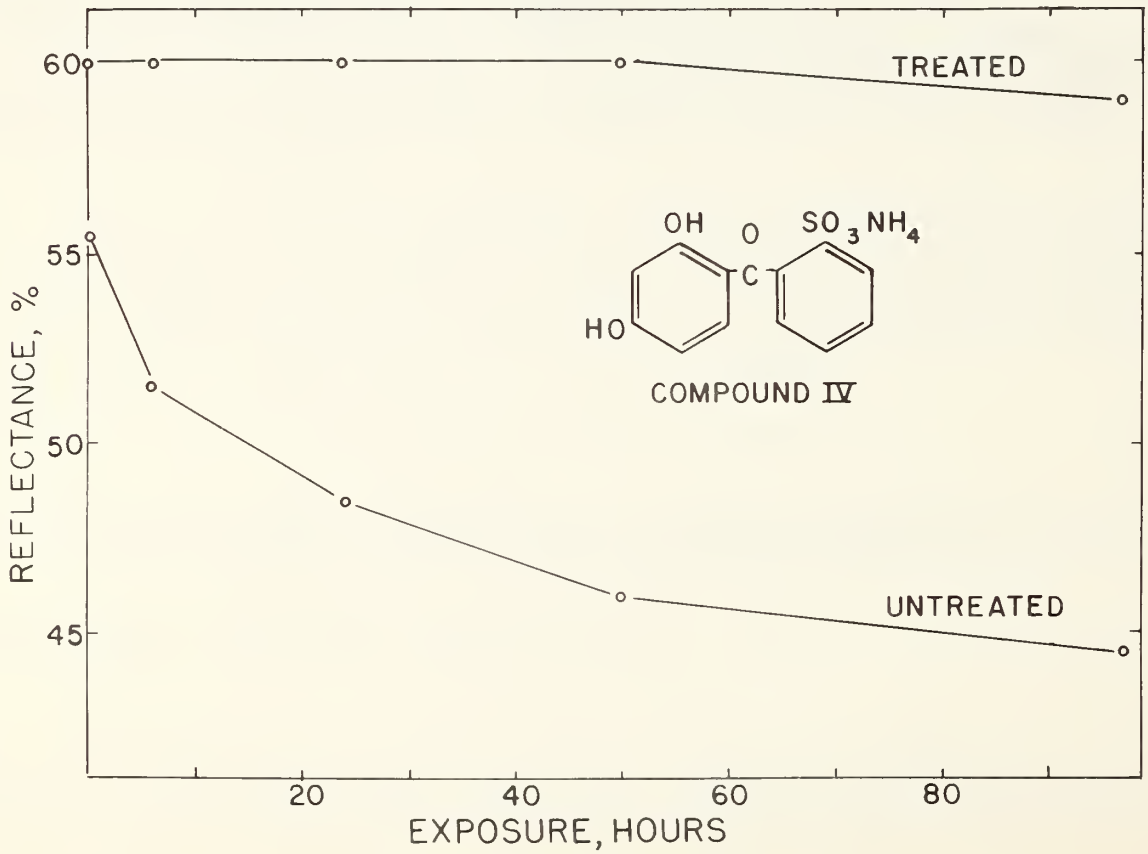

Figure 1. Prevention of ultraviolet yellowing of wool flannel by impregnation with a sulfonated hydroxybenzophenone.

a protective effect that was maintained even after extended exposure to ultraviolet light that produced substantial yellowing, shown by decrease in reflectance, of an untreated sample. The protection is relatively stable to laundering. Further work is in progress.

Resistance to chemical attack, (a) formaldehyde. Work reported by Reeves and his colleagues at the Southern Utilization Research and Development Division on the formaldehyde treatment of cotton led to reexamination of some effects of this reagent on wool (see report by W. A. Reeves, page 68). Under certain conditions formaldehyde greatly lowered the alkali solubility of wool but increased the acid solubility. However, Mayo Walden, in the Wool and Mohair Laboratory, has found conditions (for example those given in Table 1) under which both acid and alkali solubilities are very substantially decreased.

Table 1. Formaldehyde treatment of wool top.

Wool taken, dry weight	2-1/4 g.
Treating solution	
Formaldehyde (40% aqueous)	10 ml.
Hydrochloric acid (37% aqueous)	10 ml.
Sodium chloride	23.5 g.
Water	80 ml.
Reaction conditions: 5 hours at room temperature.	
Alkali solubility, decrease from control: 75 percent.	
Acid solubility, decrease from control: 58 percent.	
Weight change during treatment: 4 percent increase.	

Resistance to chemical attack, (b) isocyanates. Research is continuing on applying isocyanates for chemically modifying wool since exploratory work shows such chemically treated wools to be more resistant to acids, alkalies, and bleaches, and in some instances more resistant to felting shrinkage. Resistance of isocyanate-treated flannel to hypochlorite is illustrated in Figure 2. Much earlier work was done with pyridine as a solvent. Dimethyl formamide and dimethyl sulfoxide have recently been found preferable to pyridine in greatly speeding the reaction as well as in cost and odor (N. H. Koenig, "Isocyanate Modification of Wool in Dimethyl Sulfoxide," accepted by Textile Res. Jour.). As a result, it is now possible to get significant uptakes in only a few minutes at 105° C.

A property of immediate interest is resistance of treated fabrics to dyeing. Figure 3 shows experimental goods woven with untreated warp and alternate stripes of untreated and treated fill. The dye resistance is evident in the pattern resulting when the piece was dyed. To illustrate further possibilities, a 15% uptake of a diisocyanate produced a fabric which, although undesirably harsh, was shrink resistant, wrinkle resistant, and rapid drying.

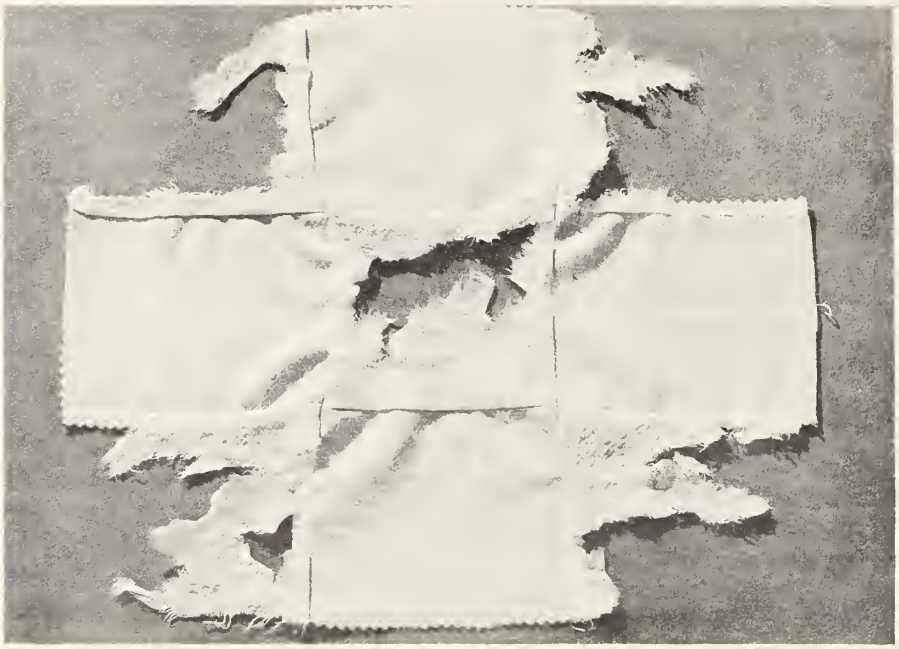

Figure 2. Effect of treating wool flannel with an organic isocyanate in lessening chemical attack. A sample made up of alternately treated and untreated squares is shown as prepared and after exposure to aqueous sodium hypochlorite (5% active chlorine) at room temperature for 8. After 10 minutes only the treated squares survive.

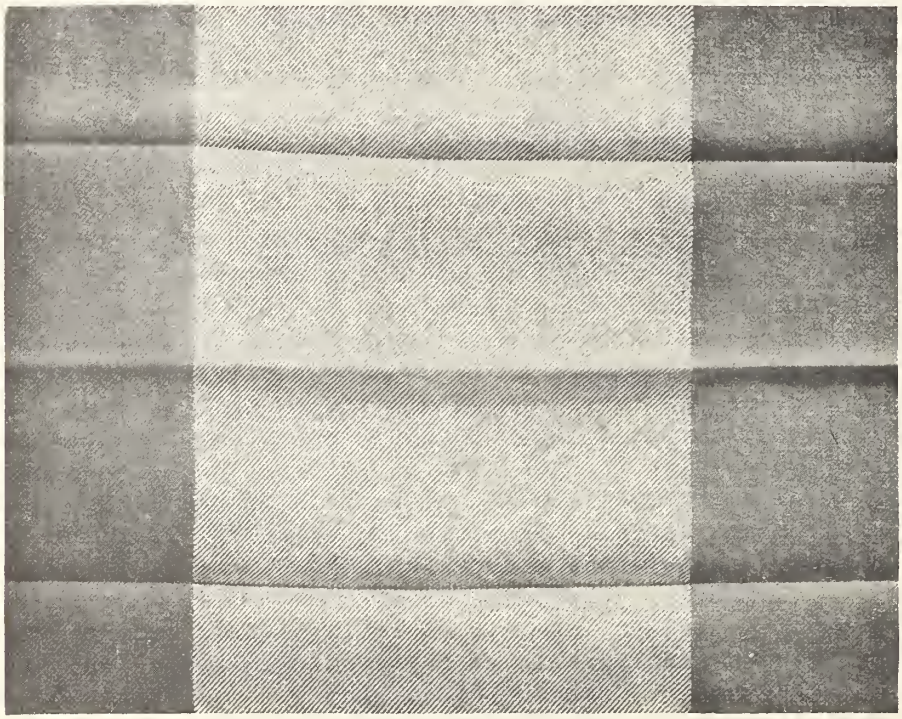

Figure 3. Dye-resist effect of treating wool with an organic isocyanate. Use of alternating sections of treated and untreated yarn for filling results in a striped pattern after dyeing.

Figure 4. Durable crease produced by ethanolamine. Pressed, creased fabrics are shown after wetting out in water. The fabric on the right, pressed in the presence of ethanolamine, retains its crease.

Chemical compaction of felts. Starting with the observation that wool fibers supercontract in hot dimethyl sulfoxide, Dr. Koenig has shown the feasibility of greatly increasing the firmness and density of felts by heating for a short time in this reagent. ("Action of Dimethyl Sulfoxide on Wool," N. H. Koenig and R. A. O'Connell, Textile Res. Jour. 30: 712, 1960.) Although individual fibers are weakened by the treatment, the treated felt is stronger than before the treatment because of the consolidation effected. It is possible that the wet strength may be improved by further modification of the process.

Durable creasing. Many processes have been described for imparting more or less durable waves or creases in keratin fibers. The better known reagents for this purpose generally cause objectionable odors during processing or when the material is wetted again. Such odors can be avoided by use of solutions of (mono)ethanolamine in water, which can be used to give durable creases resistant to wetting (Figure 4). (See report by Koenig, Wasley, and Pardo, "Setting Wool Textiles with Ethanolamine," Textile Res. Jour. 30: 901, 1960.) For such results, the amount and concentration of ethanolamine solution and

the pressing time should be adjusted to the fabric. A 1/4% solution and pressing time of 30 seconds may suffice to give a good durable crease in a worsted flannel. A 1% solution would be recommended for most suiting fabrics. The solution is sprayed on to give a weight increase of 30 to 40%. The cloth is then pressed in a steam press one minute: 30 seconds with steaming, 30 seconds without.

By adhering to suitable conditions, yellowing of the goods is avoided. Table 2 shows, by comparing solubility tests in acid and alkali, that there is no important chemical damage according to these criteria. The pH of the fabric after treatment is 8 to 8.5. Absence of fiber damage is confirmed by breaking tests, which show, incidentally, that there is no increased tendency to break at the crease. We believe that this process has commercial value.

Table 2. Effects of pressing treatment with ethanolamine on properties of wool flannel.

Ethanolamine concentration, %	Breaking load, lbs.	Samples broken at crease	Alkali solubility, %	Acid solubility, %
0.25	36.0	1 of 4		
0.5	37.3	2 of 4		
1.0	34.0	0 of 4	13.0	16.4
None	37.6	1 of 4	11.8	14.9

Samples were steam-pressed 30 seconds (for tensile tests) or 60 seconds (for solubility tests). Alkali solubility was measured after treatment with 0.1N sodium hydroxide at 60° C. for one hour, according to Harris. Acid solubility was measured after treatment with 5N hydrochloric acid at 60° C. for one hour, slightly modified from Zahn's conditions.

Discussion of Dr. Wasley's Report

Question: In the durable creasing treatment, is the fabric treated wet?

Wasley: The solution of ethanolamine is applied to the air-dry fabric, enough to give a 30 to 40% increase in weight. The wetted fabric is then pressed.

NEW INSTRUMENTS FOR EVALUATING WOOL PRODUCTS

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Development of wash-and-wear textile products is hampered by lack of objective criteria for judging fabric neatness. Accordingly, instruments have been made that measure properties by means of which one can define and determine wrinkledness and recovery from wrinkling, sharpness of creasing and crease stability, handle, and fuzziness after laundering. In addition, effects of textile mechanical processes such as combing and twisting can be observed. An electric analog computer has been assembled to derive data of interest from stress-strain measurements of fibers, yarns, and fabrics. This is also used in measuring some of the other properties mentioned.

Means proposed for defining wrinkledness and recovery from wrinkling. The American Association of Textile Chemists and Colorists (AATCC) has tentatively adopted a set of photographs of fabrics of varied wrinkledness (AATCC, Technical Manual and Yearbook, page 148, Tentative Test Method 88-1960). Test fabric can be assigned a numerical rating by comparison with these standards under specified lighting. However, the smoothest standard is too wrinkled for wash-and-wear acceptance. The specified lighting conditions are too harsh. For these and other reasons the test is likely to be revised.

Hunter and Lofland describe an Optical Wrinklemeter in which light strikes the test fabric at a low angle ("Optical Wrinklemeter," Amer. Dyestuff Reptr. 48: 54, 1959). The intensity of light reflected or scattered in different directions is compared by means of two photocells. However, different fabrics produce different patterns because of construction and color. Such factors confuse the interpretation of wrinkledness.

Another low-angle projection method has been developed at the du Pont laboratory. (Bercaw, "New Developments in Evaluating 'Wash-Wear' Performance," Amer. Dyestuff Reptr. 49: 37, 1960.) Light from a narrow slit impinges on the fabric and the distorted line is photographed from above. The amplitude of the distortions is analyzed mathematically. Details are discussed below.

Cluett and Peabody have developed an Electronic Smoothness Evaluator that may be leased by users of their Sanforized Plus process. In this instrument a beam of light grazes the surface of the test fabric laid over a roll. The silhouette is projected to a pair of photocells, the signal from which is directly related to the slope of the wrinkle. As the roll rotates, the signal is integrated, giving a measurement related to wrinkledness.

Measurement of crease angle recovery by the Monsanto method (AATCC, Technical Manual and Yearbook, page 165, Tentative Test Method 66-1959) has been approved by the AATCC. It seems to evaluate wrinkle recovery well for many fabrics of cellulosic fibers but is unsatisfactory for wool. However, even for cellulosic fabrics the results may not apply to conditions of use, which result in random, rather than unidirectional, folding and loading.

The use of average fabric height as a measure of wrinkledness, as reported by Hebeler and Kolb, is considered unreliable (Textile Res. Jour. 20: 650, 1950).

For fuzziness, Harris Laboratories compares washed samples with standards draped over a back-lighted board. Fabrics are said to be hard to differentiate by this method.

A turntable wrinkle recorder and evaluator. Wrinkledness measurements have been attempted by a quite different method in our Wool and Mohair Laboratory. Figure 1 shows the principle and Figure 2, a photograph. The test fabric is placed on a precision turntable rotated at 1/2 r.p.m. A light counterbalanced arm with a glass tip rides on the fabric at 4 inches from the center and is attached to a microtorque, high-resolution potentiometer. As the tip follows the fabric contour, its motion produces in the potentiometer a voltage proportional to fabric height. This voltage, recorded against time, constitutes a record of the wrinkledness of the fabric. In order to get an index of wrinkledness, the voltage is fed into an analog computer. Figure 2 shows the whole setup.

The hardest problem in evaluating wrinkling is to devise a mathematical formula that will agree reasonably with subjective judgments. Dr. Bercaw (cited above) has proposed that the wrinkle contour be approximated by a series of isosceles trangles, and that the wrinkle severity be evaluated as proportional to the product of the sum of the slopes of these triangles times their number. In our experience this formula fails to discriminate some fabrics that obviously differ in wrinkledness. Dr. Handy (Dr. Bercaw's associate) has suggested using the area under the contour record instead of the sum of the slopes. A limited number of tests suggests that this formula is more reasonable.

Dry and wet wrinkle recovery of resin-treated fabrics. To show the usefulness of wrinkle evaluation with this apparatus, Table 1 gives wrinkle indexes defined as suggested by Dr. Handy for fabrics finished with epoxy-polyamide and IFP polyamide resins, together with the untreated fabrics. Results are given (part A) for dry recovery from wrinkling after compression in a beaker for 10 minutes under a 4-kilogram load. All fabrics recovered rapidly. After 17 hours the IFP-treated fabric was practically unwrinkled, while the epoxy-polyamide-treated fabric was a little more wrinkled than the untreated fabric. The measurements agree with panel ratings.

Wet recovery was also measured (part B of Table 1). The fabrics were wetted out in detergent, squeezed by hand or centrifuged, and tumble-dried or drip-dried. Tumble drying is seen to give a smoother fabric than drip drying. Both resins have good flat setting effects, but the IFP treatment appears superior.

Evaluation of creasing. The system used for wrinkle measurement is readily adapted for determining the sharpness of a crease. A platform moving at 1-1/2 inches per minute in a straight line is used instead of a turntable, with the crease laid at right angles to the direction of travel. A record is obtained of the crease contour. The computer can be programed to give a value for the crease sharpness if a suitable formula can be provided. The three

Table 1. Wrinkle recovery of wool flannels.

Part A: Dry recovery after compression for 10 minutes in a beaker under a 4 kg. load

Sample	Time of recovery from wrinkling, min. 10	30	60	1,050	Wrinkledness index	Panel rating
	Area under contour, arbitrary units					
Untreated	10.2	1.7	0.7	0.5	10.7	2
IFP-finished	15.1	2.5	1.8	0	10	1
Epoxy-finished	14.2	3.2	2.7	0.8	20	3

Part B: Wet recovery after wetting out and drying

Sample	Wrinkledness, arbitrary units Drip-dried	Tumble-dried
Untreated	139	100
IFP-finished	77	20
Epoxy-finished	109	55

measurements (Figure 3) used to define sharpness of crease are: (1) the crease height, H; (2) radius of curvature, r, estimated by a circle closely approximating the contour at the apex; and (3) Θ, the supplement of the angle formed by extending tangents to the crease contour at the points where it departs from the matching circle.

Twenty-four fabrics were experimentally creased with ethanolamine under conditions giving a wide range of sharpness. These were rated by a panel, and crease contours recorded instrumentally were evaluated in terms of the three parameters H, r, and Θ. Crease sharpness indexes $\frac{H\Theta}{r}$ and $\frac{\Theta}{r^2}$ tended to agree with the panel's judgment (Figure 4).

Evaluation of textile and fiber properties by sound analysis. Fabrics or fiber masses rubbing against themselves or other chosen surfaces under controlled conditions make a noise that can be measured and analyzed and used to characterize the material. Appropriate apparatus, diagramed in Figure 5, has the test fabric mounted over a microphone and rubbed by a moving, rotating head. The electric signal generated in the microphone by the noise is amplified and fed into a wave analyzer, which permits measuring the signal intensity as a function of frequency. The sound intensity spectrum can then be plotted. Slight modification permits measurement of noise produced in bulk wool or in slivers. Four representative applications of sound analysis are illustrated.

The sound intensity spectrum is expected to be influenced by fiber properties such as flexibility, elasticity, density, diameter, and surface friction, and by properties of the assembly such as the fiber spacing, distance between contacts, yarn size and twist, fabric construction, and surface contour. Many of these properties are also related to the hand of a fabric. It is therefore not surprising that changes in finishing that affect the hand also

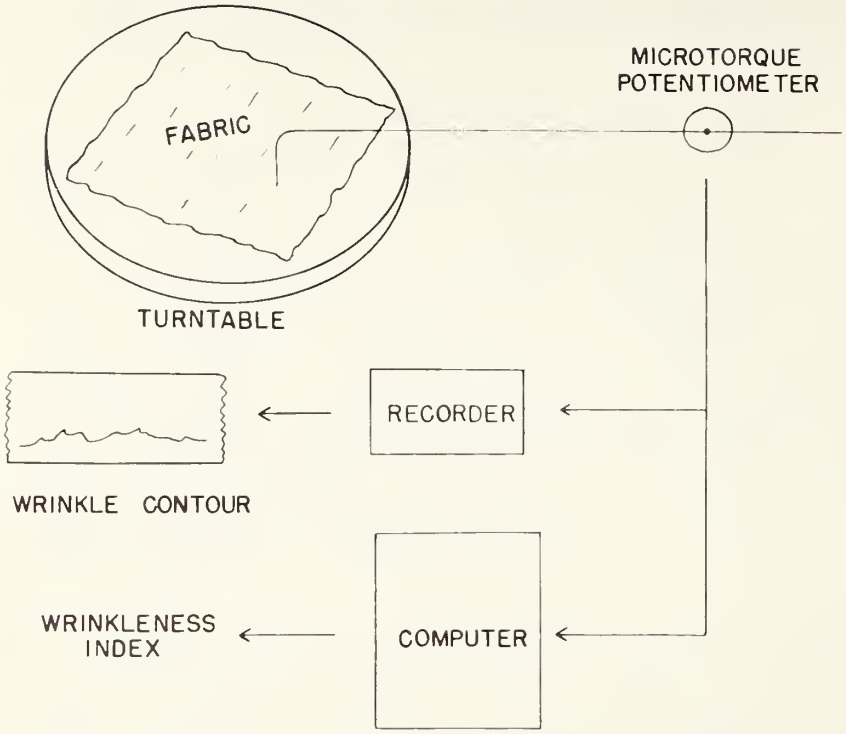

Figure 1. Principle of fabric wrinkledness evaluation. Fabric height is sensed mechanically, amplified electrically, recorded, and evaluated by an electric analog computer.

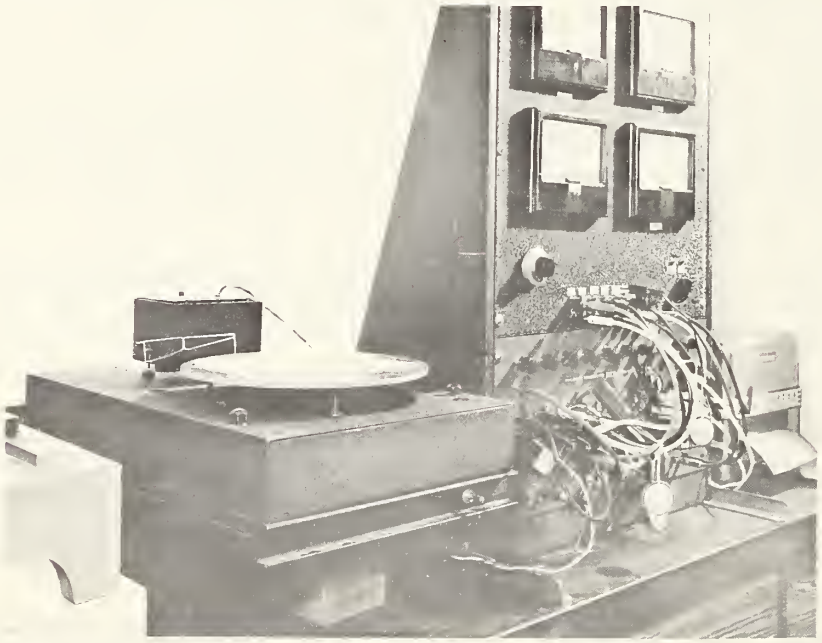

Figure 2. Apparatus for evaluation of fabric wrinkledness, showing relation of turntable, sensing element, computer, and recorder. The computer is programed to yield a mathematically defined index of wrinkledness. Turntable rotates at 1/2 r.p.m. Rounded tracing point is glass to minimize friction. Movement of tracing point is converted to electric signal by a microtorque potentiometer.

Figure 3. Fabric contour record of a crease, indicating parameters proposed to define crease sharpness. H: height of top of crease above level surface. r: radius of curvature of circular arc approximating top of crease contour. θ: angle subtended by circular arc approximating top of crease contour between the limits at which the true curve departs from the matching circle.

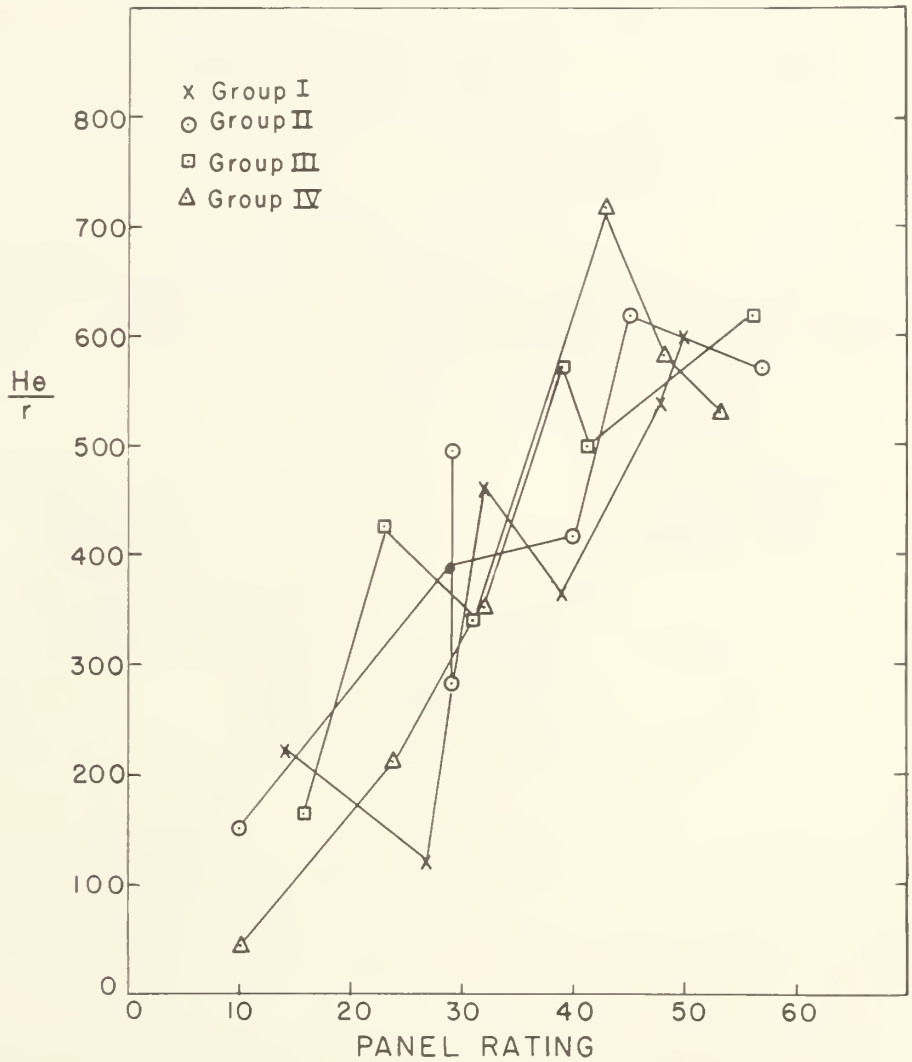

Figure 4. Comparison of a crease index with subjective rating of creased fabrics. Twenty-four creased fabric samples in four groups were evaluated by the arbitrary index $\frac{H\theta}{r}$. These values are compared with average ratings assigned by ten untrained judges.

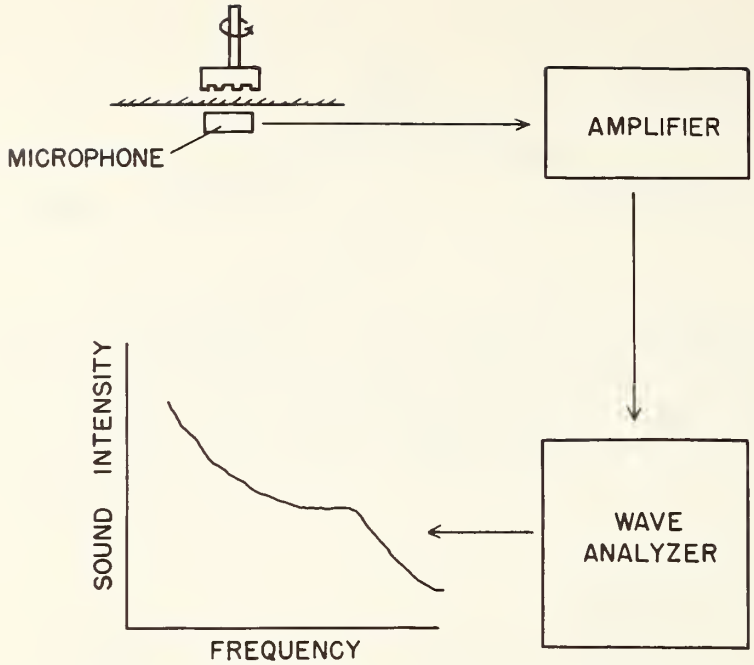

Figure 5. Principle of fiber or fabric evaluation by analysis of sound produced by rubbing. A rotating disc in contact with the fabric surface produces sound which is sensed by a microphone and analyzed in terms of intensity and frequency. Alternative mechanical arrangements are used for various fabrics or fiber in bulk.

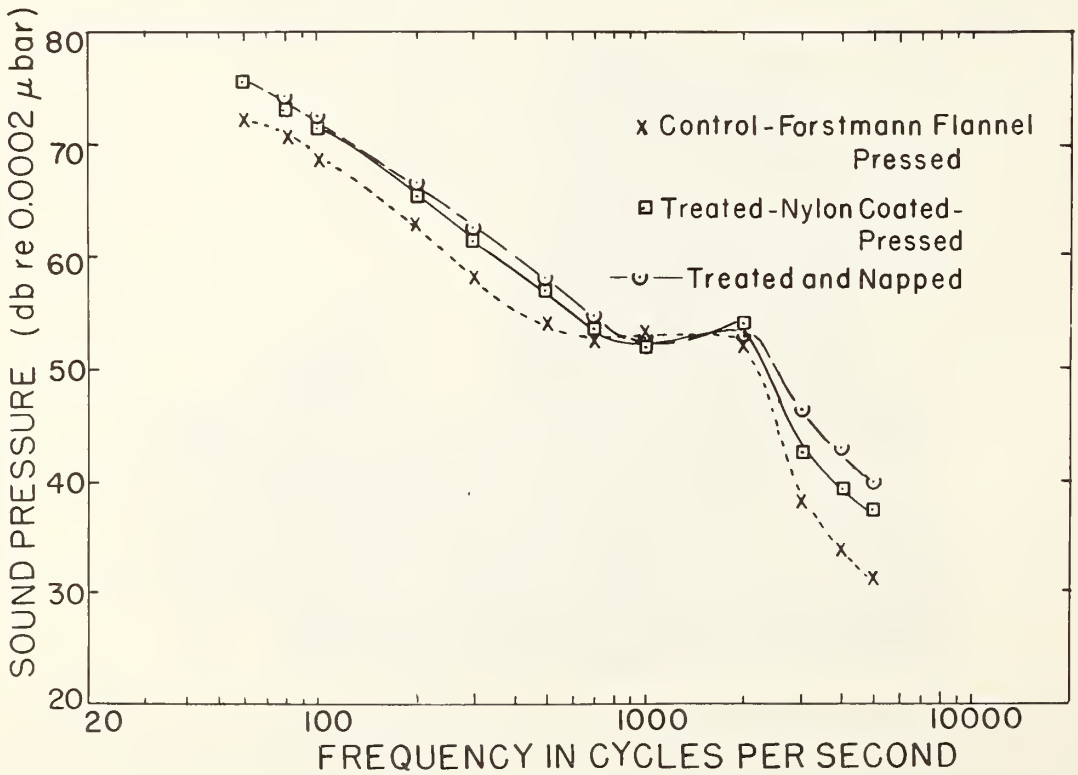

Figure 6. The effects of polyamide treatment (IFP) and napping on the sound intensity spectrum of a wool flannel.

change the sound spectrum. As an example, Figure 6 compares the spectrum of a flannel with spectra of the same material with a polyamide finish by IFP, and again after napping. Slight stiffening resulting from the treatment is associated with increased sound intensity except in the neighborhood of 1,000 cycles per second. Napping produces a further increase that may be due to a larger number of movable fiber contacts.

Effects of mechanical processing from carding through the second autoleveling after combing have also been determined, as shown in Figure 7. Sound intensities decrease regularly with the process of fiber alignment. The effect of fiber crimp was further tested by passing samples from the autoleveler (sample B) over a hot roll to reduce crimp further. Sound intensities were thereby further decreased (samples C and A) but restored to the level of card sliver by steaming.

Sound analysis can be used to compare fuzziness of fabrics by setting the rubbing head at a fixed distance from the fabric so that only fibers projecting a certain distance will make contact. A series of fabrics rated for fuzziness by visual comparison was placed in the same order by the sound intensity spectra, except for a single reversal attributed to error in the visual rating.

Table 2. Estimation of wrinkledness by sound intensity measurement. Effects of polyamide (IFP) finish and of drying procedure are shown. Two techniques for generating the sound are compared: one with blanket material and the other with a metal tube, both at fixed distance from support rather than at constant load.

Fabric and treatment		Sound intensity as millivolts, arbitrary scale	
		Blanket head	Metal head
Original worsted flannel	pressed	9	10
Original	wet out, drip dried	46	68
Original	wet out, tumble dried	22	68
IFP-treated	wet out, drip dried	14	60
IFP-treated	wet out, tumble dried	12	46

Finally, a similar technique with rubbing head set at a fixed distance can be used to measure wrinkling or mussiness, which causes parts of the fabric to stand away from the supporting surface. This procedure appears sensitive to small degrees of wrinkling, so that it has special promise for wash-and-wear performance. Table 2 shows electrical outputs produced by sound with two different rubbing heads on a worsted flannel, on the same fabric wetted out and drip dried or tumble dried, and on the same fabric finished with polyamide by IFP, wetted out and drip dried or tumble dried. The fabrics are clearly ranked by the sound intensity in order of wrinkledness. It is evident that the treatment has lessened the effects of wetting out and drying and that in this case, drip drying leaves a more wrinkled surface than tumble drying.

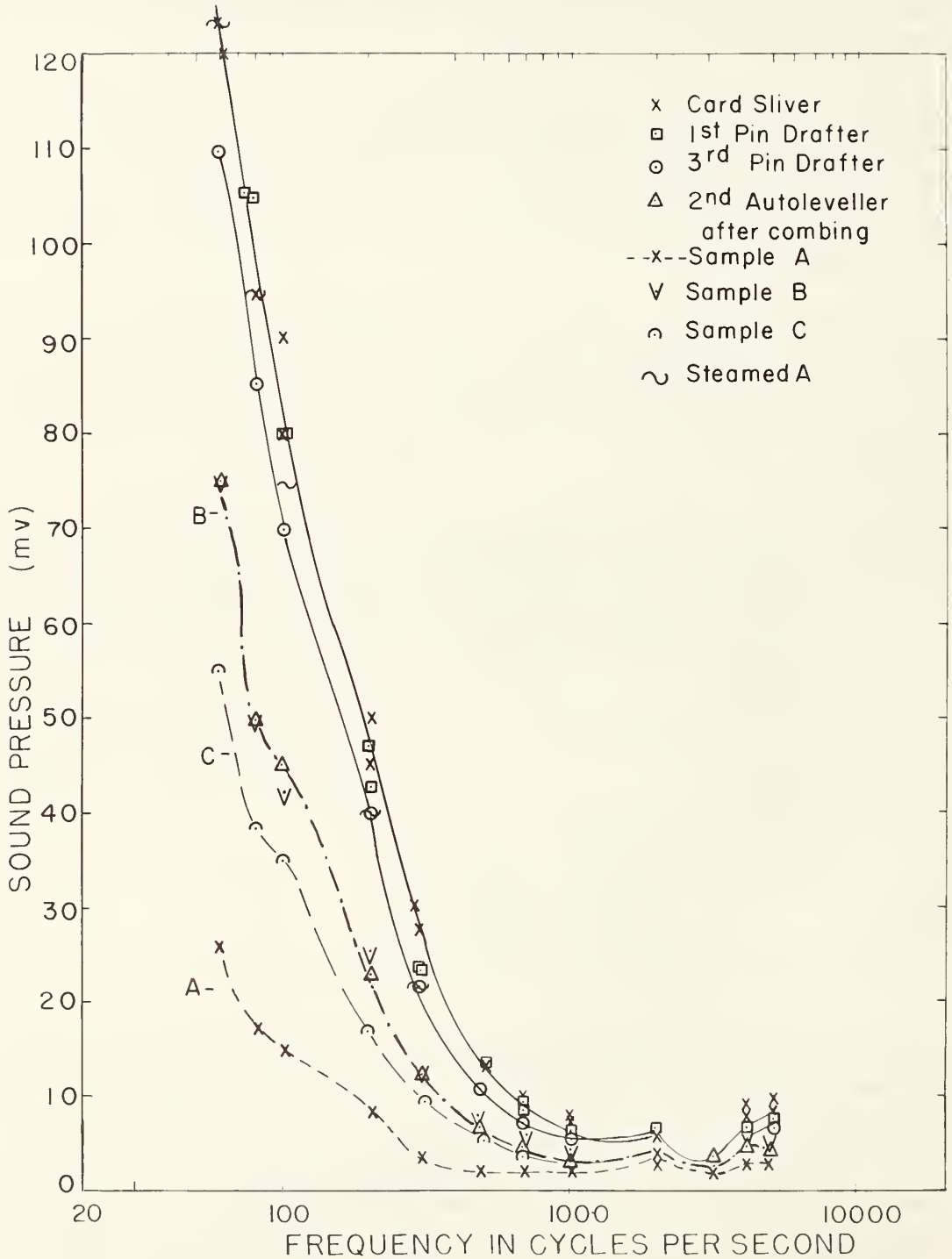

Figure 7. Sound intensity spectra of wool fiber assemblies at various stages of mechanical processing. The intensities are decreased by alignment and straightening of the fibers.

Use of an electric analog computer to help evaluate tensile properties. The value and convenience of an electric analog computer for converting measurements of wrinkles and creases into appropriate numerical indexes has been mentioned. Such a computer has also been set up to find various significant quantities from stress-strain analyses of fibers, yarns, and fabrics ("A Computer for the Automatic Processing of Fiber, Yarn, and Fabric Stress-Strain Data," W. J. Thorsen, Textile Res. Jour. 30: 827, 1960). The computer has the advantages of (1) independence of variations due to the analyst's judgment and (2) displaying the results as soon as the measurements are finished.

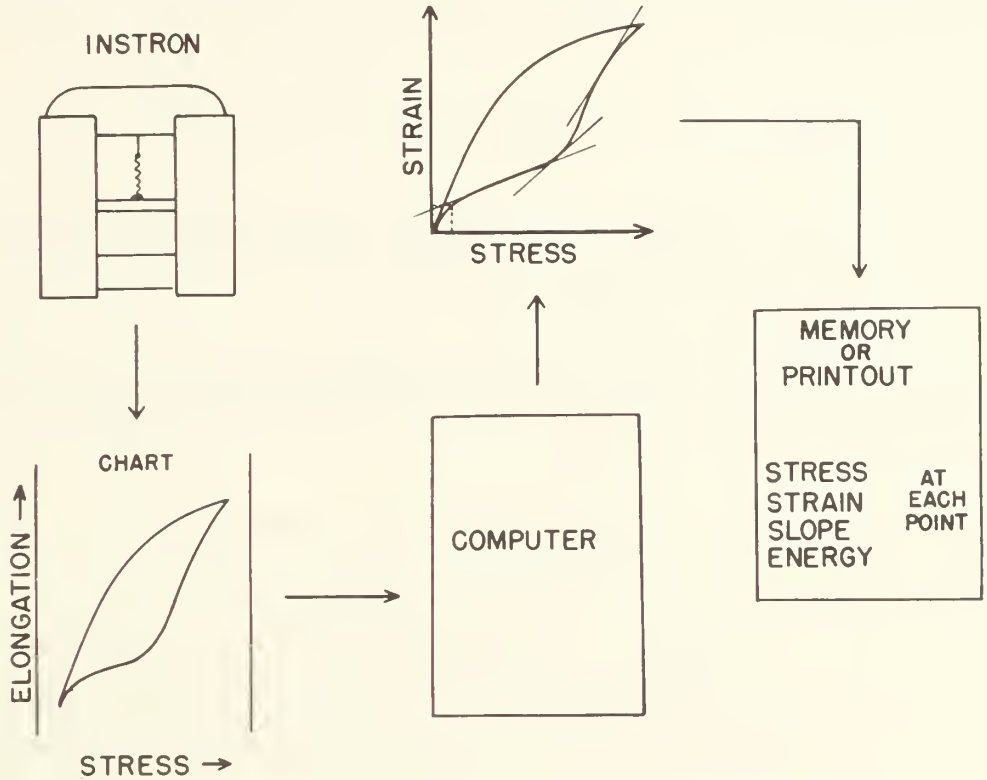

Figure 8. Principle of use of electric analog computer to evaluate stress-strain tests. The tensile tester produces a load-elongation diagram. The motion of the tester generates electric signals that the computer uses to determine first, second, and third derivatives (rates of change) of load as a function of elongation. These are combined, with appropriate constants, to produce the parameters of interest in defining mechanical properties, such as the stress at given percent elongation, Young's modulus, yield point, work to extend, work lost during cycle, or others. These desired quantities are presented as meter readings or counter readings.

The equipment is diagramed in Figure 8 as used with an Instron Tensile Tester for single fiber tests. The force and elongation mechanisms of the Instron are used to supply signals to the computer, which operates on them to produce values of Young's modulus, yield stress, and the other significant quantities as directed. The relatively inexpensive apparatus now operable has a capacity of displaying six chosen parameters as meter readings, but could be readily adapted to print these, or a larger selection, on tape.

Discussion of Mr. Thorsen's Report

Question: What is the effect of the arm weight or tracing point pressure on the performance of the turntable wrinkle tester?

Thorsen: This has not been tested in detail. However, if the weight is made too light the machine responds to fuzziness. This can be shown by comparing a wrinkled fabric and the same with the wrinkles pulled out.

Question: Is the tracing point always used at the same distance from the center of rotation?

Thorsen: This position is not rigidly fixed, but the approximate distance of 4 inches has been adopted for the tests reported.

Question: Is there any difficulty in spreading out the fabric uniformly on the turntable? That is, is a numerical rating valid for the piece of material in general or only for the particular part that encounters the tracing point? Do you have any suggestions for achieving uniformity?

Thorsen: We haven't developed any special technique for insuring uniformity. Nevertheless, the numerical values are amazingly reproducible. This suggests that the result does apply to the test piece as a whole rather than just to the part touched by the tracing point.

Question: Have you excluded optical methods from consideration?

Thorsen: No, but this mechanical method seems to have some advantage.

Comment: In studies in this field, definition of wrinkledness must be somewhat arbitrary. Optical methods may allow combined judgment of wrinkledness and other factors interpreted as mussiness. It is also evident that fabric structure and color pattern will affect subjective judgment of mussiness in a way that is inaccessible or much less accessible to instrumental measurement.

Thorsen: In developing wash-and-wear properties, however, an instrument that responds only to the geometrical differences between treated and control fabrics has special value.

Question: How do you define fuzziness, wrinkledness, and mussiness?

Thorsen: Fuzziness depends on density and length of fibers standing up from the fabric surface. Wrinkledness depends on definite regions with relatively well defined maxima not resting on the supporting surface. Disordered areas with less extensive variations and without well defined peaks are called mussy. Mussiness is sometimes considered to be due to "microfelting." Instrumental definition depends on correlation of measurements with judgments.

Comment: A perfectly sharp crease might be considered too sharp to be attractive. In this case, measurements could be at variance with consumer acceptance.

Thorsen: In such a case it would be necessary to define conditions of consumer acceptance with the help of a panel of judges.

DEMONSTRATIONS AND EXHIBITS IN THE WOOL PROCESSING LABORATORY

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Western Regional Research Laboratory
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The exhibits included (1) fabrics durably creased with ethanolamine, (2) material showing the dye resistance of isocyanate-treated yarn in a woven striped pattern, (3) diisocyanate-treated material showing resistance to hypochlorite under conditions that completely disintegrate untreated fabric, and (4) felts consolidated by treatment with hot dimethyl sulfoxide. The electric analog computer as an attachment for the Instron Tensile Tester was displayed in the testing room, which is in the process of being equipped.

Application of epoxy-aminopolyamide resin by exhaustion. Application of epoxy-aminopolyamide by exhaustion has been sufficiently promising in development that work is now in progress to adapt it to full-scale equipment. An attractive feature of the process is the possibility of partially curing the resin during dyeing; so that application, partial curing, dyeing, and washing can be done in the same equipment. As an example of current development work, 15 yards of experimental material were treated with epoxy-polyamide resin by exhaustion and then dyed. The entire process was carried out in a sample dye beck with an adjustable reel able to handle up to 60 yards of fabric.

Batch application of polyamide by interfacial polymerization. To illustrate the possibility of applying polyamide resin to made-up wool goods not suitable for continuous processing, socks were treated individually by hand dipping first in 2 percent aqueous hexamethylene diamine with 4 percent sodium carbonate (as the anhydrous salt) for about 30 seconds. Excess reagent was spun off in a Tolhurst textile centrifuge, run at 1100 r.p.m. for one minute. The socks were then hand dipped in a 3 percent solution of sebacoyl chloride in Stoddard solvent for 30 to 45 seconds. To avoid polymer formation in the equipment, the centrifuge was thoroughly rinsed with water and wiped dry before spinning off excess acid chloride treating solution. After centrifuging, the socks were then ready for washing and finishing. The centrifuge was again rinsed with water and wiped dry before use with the next batch.

Similarly treated socks take on about 2 percent of resin; they have been found to be essentially shrinkproof through at least 12 wash-and-wear cycles. Although socks were treated individually for this demonstration, work is under way on larger batch application.

Continuous application of polyamide by IFP. Treatment of test pieces 14 to 16 inches wide by dipping and padding was also demonstrated as normally used in determining conditions needed for full-width processing. For the demonstration, 2 yards of ready-to-dye wool flannel (6 ounces per square yard) was sewn to a 2-yard cotton leader. A "ten-ton" two-roll textile pad, with 72-inch rolls, 18 inches in diameter, faced with firm synthetic rubber, was used at 2 yards per minute and a roll load of about 400 pounds per linear inch for the first pass. Aqueous 1/2 percent hexamethylene diamine with 1 percent sodium carbonate (as the anhydrous salt) was applied at room temperature (80° F.) by dipping in a special narrow stainless steel trough at the left side

of the pad. Contact time in the trough was about 10 seconds. The amine impregnated fabric was taken from the squeeze rolls and started through the second part of the treatment within 2 minutes. Three percent sebacoyl chloride in Stoddard solvent was then applied by dipping at the right side of the pad in a narrow trough like that used for the diamine. As before, the time in the trough was about 10 seconds. Excess solution was removed with a somewhat lower pad load, about 300 pounds per linear inch. The fabric was then ready for washing and finishing. Resin uptake was 1/2 percent. Area shrinkage after five 75-minute accelerated wash tests was less than 5 percent.

Samples of the treated material were distributed Friday morning.

Hand and appearance of resin finished fabrics. Effects of the epoxy and IFP stabilizing finishes on hand and appearance were shown by displaying 20 treated fabrics, some with untreated portions of the same piece, for evaluation and comment. The shrinkage stability of these materials was also available. Fifty-five participants at the conference recorded judgments, which are summarized in Table 1. The small number of observers and the conditions of judgment are such as to make fine distinctions impossible. However, although substantial differences in opinion were expressed, there are no marked differences in averages for consumers as opposed to manufacturers or men as opposed to women. Most fabrics were rated acceptable or better. We like to think that this is not merely an expression of courtesy.

Figure 1. Treated fabrics displayed for judgments by conference attendants.

Table 1. Hand and appearance of shrink-resistant fabrics.

Fabric and treatment[1/]	Control[2/]	Not acc.[3/]	Acc.[3/]	Good[3/]
Grey Donegal tweed; IFP	Yes	7	32	16
Grey Donegal tweed; Epoxy	Yes	13	28	14
Dark grey worsted flannel; IFP	Yes	5	24	26
Light brown woolen flannel; IFP	Yes	8	21	26
Brown, cross-dyed sheen gabardine; IFP	Yes	0	25	30
Red woolen skirting; IFP	No	6	26	23
Grey woolen; IFP	No	3	30	22
Brown and blue woolen plaid; IFP	No	8	36	11
Olive worsted; IFP	No	5	29	21
Orange face-finished woolen; IFP	No	37	15	3
Brown worsted; IFP	No	1	26	28
Coral woolen; IFP	No	7	35	12
Yellow-brown hopsack; Epoxy	Yes	9	27	19
Orange, black, blue worsted skirting; Epoxy	Yes	23	26	6
Pink blanket; IFP (dyed and napped)	No	18	31	5
White blanket; IFP (napped)	No	18	29	7
Blue worsted flannel; IFP (1% HMDA) (dyed after IFP with acid premetallized dye)	No	4	26	25
Blue worsted flannel; IFP (1/2% HMDA) (dyed after IFP with acid premetallized dye)	No	5	17	33
Grey worsted flannel; IFP (2% HMDA)	No	3	12	33
Dyed and white Jerseys; IFP (flat bed pressed)	No	5	23	19

1/ IFP: Polyamide finish by interfacial polymerization applied by padding. Epoxy: Epoxy-aminopolyamide finish applied by padding. HMDA: Hexamethylene diamine was used throughout. Concentrations are listed for certain samples, for which the differences are of interest. All fabrics were finished by dolly-washing. A softener was applied. Most fabrics were pin-tenter-dried, pressed, and decated, except that the blankets were napped and the Jerseys dried without tentering, then flat bed pressed.

2/ Yes means that untreated fabric was available for comparison.

3/ Numbers of persons judging the fabric as, respectively, not acceptable, acceptable, and good. No instructions were given as to criteria for evaluation.

WOOL AND BLENDED FABRICS

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Thank you for the invitation to attend the Wool Finishing Conference. I was privileged to be here at the dedication of the New Wool Laboratory on November 2, 1959, and bring the greetings of the National Wool Manufacturers. It makes me very happy to come out to sunny California again, especially when I look back and visualize the pile of snow I left in New Jersey.

The question is often raised: Why was the Wool Pilot Plant established in the West? Let me give you the story. My first visit to this Laboratory was in the Spring of 1948, to discuss the Wool Research Project started at Princeton. When I was elected a member of the Wool Advisory Board in 1951, I became more closely associated with the work and especially with the people under Dr. Copley and Dr. Lundgren. In the early 1950's the members of the Wool Advisory Board became aware of the need of a Wool Pilot Plant similar to the Cotton Pilot Plant in New Orleans. Even though political as well as manufacturing interests were strongly recommending a pilot plant in the East, and after fruitless efforts made by the Technical Committee and the Wool Advisory Board to concentrate the government wool work at one place, either at Beltsville, Maryland, Denver, Colorado, or here at Albany, we finally ended up with the following recommendation at the meeting of October 10, 1956:

> "1. Recommended strongly and unanimously that a pilot plant be established, capable of processing small quantities of wool, even single fleeces, from the raw state to a finished fabric, and also designed to handle larger quantities of wool continuously. This facility is needed to evaluate chemical modifications of wool fibers and other treatments that may be applied to wool in varying steps of processing. The location of the pilot plant is immaterial but, insofar as practicable, this facility should be made available, under suitable arrangements, to researchers, in addition to those of the Department of Agriculture, who are working on wool problems."

Although the location was not specified, it was clear that the pilot plant should be at a place where the most able people and the most enthusiastic wool researchers in this country were located and even the enemies had to agree that there was no better group than the one under the leadership of Dr. Harold Lundgren. What we have heard and seen today is proof that the confidence we had in this group was more than justified.

As a collaborator of the Western Utilization Research and Development Division, I feel it my duty more than ever to make my contribution in challenging and encouraging Dr. Lundgren and his group in their future work.

There are only very few mills left in this country which work with wool only. The Pendleton Woolen Mills, under the leadership of the Bishop family, are the faithful ones here on the West Coast.

Fleece and pulled wools represent probably less than 50 percent of all the materials used by the woolen mills in this country today and it is especially the inroad of synthetic fibers which we must acknowledge.

In the last few years a change has taken place in the field of wool research, endeavoring to endow wool with fiber properties already possessed by the synthetics. I have to agree with Professor Whewell of Leeds, that recent developments in wool finishing have been highly stimulated by successful developments in the man-made fiber industry. However, we should not forget that a great effort is still being directed by the man-made fiber producers towards endowing man-made fibers with the same characteristics of the God-made fibers and specifically wool. The most striking example is the new Orlon 21 - Sayelle. This fiber consists of a bicomponent structure resulting in a three dimensional spiral crimp very closely resembling fine wool.

The nature and pattern of future textile fiber competition between synthetic fibers and wool is under continuous study by the wool growing countries and specifically Australia. In a recent paper, "Competition Between Fibers" (G. O. Gutman and M. Fead, July 1959), published by the Australian Bureau of Agricultural Economics, the authors point out that the recent concept of a marriage of the fibers is not based on a union between equal partners and may, as a result, prove unstable.

The main advantage synthetic fibers have over wool is that their supply can be adjusted readily to changes in demand, thereby giving stabilized price structures. The stabilized price of the synthetic fabrics is one of the most difficult properties with which wool fabrics must compete. The survey continues that there can be little doubt that sharp fluctuation in wool prices and the attendant difficulties in planning provide an incentive to textile manufacturers to substitute synthetic fibers for wool. This may have the effect of narrowing the fluctuation in wool prices. The supply of wool is rising more slowly than the world demand for textile fabrics, thus forcing a greater use of synthetics. This will have the effect of guiding wool into those end uses for which it is particularly well adapted and for which few satisfactory substitutes exist. The survey also is critical of the way in which wool has been promoted to the public. Whereas wool promotion has sometimes had the appearance of dealing with synthetic fibers by ignoring them, manufacturers of synthetics have taken the lead in examining their fibers and blends with wool and in studying their adaptability for new end uses. Another important aspect is that the textile industry itself is becoming more highly integrated in the United States and in some other countries. This means that any wool promotion campaign which "deprecates or ignores" blending fibers is probably doomed for the reason that the major wool processors are already heavily engaged in the production of synthetic blend fabrics.

It appears that synthetic fiber production may well expand at the expense of other fibers by depressing the selling prices of the other fibers or by slowing down their production. The survey concludes that, "One cannot with certainty assert that the future of wool is assured. Small changes in the rate

at which total demand for textiles is expanding and quite minor shifts in the pattern of usage of the various fibers can result in major changes in the demand for wool."

The results of this survey present the wool fiber technologist, the synthetic fiber technologist, and the textile industry with major challenges which, of course, could alter its conclusions considerably.

Dr. Lundgren and coworkers, you have given proof today that you are more than willing to take up this challenge. Keep up the good work and you can be assured that the wool growers and the wool manufacturers will stand by you.

MILITARY PREFERENCES IN FINISHED WOOL FABRICS

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Dr. Kennedy first reviewed the evolution of military uniforms in Europe and America, which has been dictated by available materials, state of technological achievement, and prevailing styles. The United States now has separate service and field uniforms, the latter designed strictly for serviceability and protection against natural and enemy-imposed environments. Use of wool is shown in Tables 1, 2, 3, and 4 comparing the present uniforms with the preceding. The moderate decrease from 21 to 18 pounds results mainly from eliminating the wool overcoat as an article of combat clothing and more efficient use of wool as an insulating material in the present combat clothing. A reduction in the relative proportion of coarse wool and an increase in that of fine is shown in Table 5.

Table 1. Army winter combat clothing.

World War II	1961
Underwear, shirt & drawers, 50% wool/50% cotton	Underwear, shirt & drawers, 50% wool/50% cotton
Wool shirt, 80% wool/20% cotton	Wool shirt, 85% wool/15% nylon
Wool socks	Wool socks, 50% wool/25% cotton/25% nylon
Cotton field jacket	Cotton field jacket with wool frieze liner
Wool jacket	
Wool trousers	Wool trousers, 85% wool/15% nylon
--	Cotton trousers with wool frieze liner
Wool overcoat	Cotton overcoat with wool liner
Wool gloves	Leather gloves and wool inserts
Wool dress cap	Wool dress cap
Wool knit cap	Cotton cap, wool pile & wool/cotton flannel lining
Wool sweater	--
Wool scarf	--

Table 2. Amount of wool in the Army winter combat clothing.

	WW II Lbs. of wool[1/]	1961 Lbs. of wool[1/]
Underwear, shirt & drawers	1.10	1.29
Wool shirt	1.26	2.29
Wool socks	.13	.09
Cotton, field, jacket with pile liner	1.82	--
Wool jacket (Eisenhower)	2.70	--
Cotton, field, jacket with wool frieze liner	--	1.80
Wool trousers	2.40	2.04
Cotton trousers with wool frieze liner	--	1.57
Wool overcoat	8.80	--
Cotton overcoat with wool liner	--	1.34
Gloves	.16	.11
Wool knit cap	.17	--
Cotton, field cap, wool pile & wool/cotton lining	--	.17
Wool sweater	1.56	--
Wool scarf	.57	--
TOTAL	20.67	10.70

1/ Estimated pounds of clean wool required to produce the item including estimated waste in manufacture.

Table 3. Amount of wool in the Army service uniform.

	World War II[1/]	1961[1/]
Jacket, wool OD (Eisenhower)	(2.70)	--
Trousers, wool OD	(2.40)	--
Shirt, wool OD	(1.76)	--
Cap, wool, garrison	.25	.25
Coat, wool, AG-44	--	4.44
Trousers, wool, AG-44	--	2.19
Cap, wool service	.22	.22
	.47	7.10
TOTAL	(6.86)	

1/ Estimated pounds of clean wool required to produce the item including estimated waste in manufacture.

Table 4. Amount of wool in the Army winter combat and service uniforms.

	WW II Lbs. of wool[1/]	1961 Lbs. of wool[1/]
Winter combat uniform	20.67	10.70
Service uniform, AG-44	.47	7.10
	21.14	17.80

1/ Estimated pounds of clean wool required to produce the item including estimated waste in manufacture.

Table 5. Amount of wool by grade used in Army winter combat clothing.

Grade	WW II Lbs. of wool 1/	1961 Lbs. of wool 1/
64's	--	--
62's	1.10	9.65
60's	6.83	3.24
58's	.88	--
56's	.86	.97
54's	--	--
52's	--	--
50's	3.55	.67
48's		--
46's	7.92	--
44's		--
42's	--	--
40's	--	--
38's	--	--
36's	--	3.37
TOTAL	21.14	17.80

1/ Estimated pounds of clean wool required to produce the item including estimated waste in manufacture.

Present Army cold weather combat clothing has been developed, based upon scientific data from the fields of physiology, physics, and textile technology. Three major principles have been adopted: (1) achievement of insulation efficiency with minimum weight by effective use of air entrapment; (2) use of a water- and wind-proof outer shield fabric; (3) adoption of a layer system of clothing to accommodate the wide temperature range over which cold weather clothing must be worn and to adjust for changes in activity levels, loss of insulating value, or loss of comfort. Other requirements affecting design are the need for minimum weight and complete launderability without excessive dimensional change. These principles led to the adoption of cotton wind- and water-proof jackets and trousers with underlayers primarily of wool for insulating value.

The main articles of wool in the present cold weather clothing are the following: (1) The very popular lumberjack-style shirt, designed to be worn either in or outside the trousers. This is made of 16-ounce heavily napped shirting using 45 percent 62's wool with 40 percent noils, strengthened by addition of 15 percent nylon staple. The fabric must be treated for shrinkage resistance. Most of it is chlorinated. In tailoring, residual relaxation shrinkage of 5 percent in each direction is allowed for, in addition to felting shrinkage of 5 percent (warp) and 3 percent (fill). Sleeves and collar are laid out across the warp. Even so, overall shrinkage is excessive. A requirement that the fabric be treated by compressional shrinkage is now being considered.

(2) A 12-ounce, two-faced, napped (sanded) frieze made of coarse wool will soon be adopted as a good approach to an effective, minimum-weight insulation which drapes and launders well.

(3) Serge trousers of 18-ounce wool with 15 percent nylon for improved wear have been developed for use either as extra insulation beneath the cotton field trousers or as semidress trousers in the field.

(4) Underwear (like socks) has always been a blended item. A half-wool, half-cotton combination gives optimum insulating value with minimum skin contact and optimum softness, moisture absorption, ease of laundering, and freedom from static. The combination is not equaled by any single fiber or other blends. Heat transfer, including that due to the evaporation-condensation cycle is minimal, partly because of the ability of wool to absorb much water without a liquid phase forming, and partly because of low wicking tendency. Finally, wool/cotton combinations (best with 80 percent wool) form durable residual chars when exposed to temperatures of 700-800° F., more so than either pure constituent.

(5) Socks are made with stretch nylon as reinforcing yarn and to decrease the number of sizes needed (from 11 to 3). All-wool worsted terry yarn from 56's wool provides resilient area on the bottom of the foot. Socks stand out as an example of complex fiber combination utilizing unique properties of several different fibers to best advantage.

Wool is not unchallenged as an insulating material. Alan Woodcock and Lyman Fourt have conducted a simulated metabolic test (unpublished) in which a synthetic material, lighter in weight than wool, permitted greater heat loss than wool during a period of activity, but less during a following period of inactivity during which sorbed moisture from the first period was still being evaporated from the wool. The possibility of selective insulation emphasizes need for careful evaluation of actual fiber properties and use requirements.

Blends with synthetics have been considered in detail. Apart from the uses of nylon already mentioned, the only blend shown to give better performance than all-wool is a 40 percent wool and 60 percent polyester blend in the fabric for officers' summer uniforms. However, level-dyeing this mixture is still a problem. (Textile Series Report No. 108, Quartermaster Research and Engineering Center, Natick, Mass. 1959.)

The most significant military finishing requirement is shrink resistance sufficient for field laundering. Another major requirement is dyeing to shade, with color fastness. Permanent creasing and wrinkle resistance are important in uniforms. Of three processes found to be technically satisfactory, two give unpleasant odors when the goods are wet out. The USDA ethanolamine treatment appears to lack this characteristic and is under study.

Certain other properties would be valuable if they were available, such as permanent mothproofing. Present practice is to apply DDT in a sponging operation, which protects the garment until it is drycleaned. A simple process giving permanent protection would be useful. An effective water-repellent treatment would be usable in the officers' dress overcoat. In chemical finishing, additive processes appear preferable because they are less likely to impair fabric strength. All chemical treatments must be carefully planned to assure retention of serviceability.

On its merits, wool is an excellent fiber for military use, but one cannot accept without question that its properties always best serve the needs of man. Other fibers or other means may be invented that will do required jobs even better. This is the real challenge to those concerned with wool research--to see that wool does not fall behind in the present, ever-increasing pace of textile development.

Discussion of Dr. Kennedy's Report

Question: What is the Quartermaster's attitude toward dieldrin for permanent mothproofing? (Cf. "The Mothproofing of Wool with Dieldrin," M. Lipson and J. R. McPhee, Textile Res. Jour. 28: 679, 1958; "Durable Mothproofing of Wool--New and Inexpensive Methods Using Dieldrin," Wool Science and Technology Report No. 2, The Wool Bureau, New York, 1958.)

Kennedy: In spite of acceptance of use of dieldrin elsewhere, the Army has not yet adopted this treatment because of toxicity hazard.

Question: Concerning effective water repellents: can't silicones be used?

Kennedy: These have proved neither sufficiently water repellent nor durable in our experience.

Question: What about Scotchgard?

Kennedy: The effectiveness of some of the fluorocarbon treatments we evaluated was lost during the first drycleaning. These are therefore not economical for wool. Five durable types of water repellents have been approved for cotton, but none was truly permanent because all became masked by soap deposits from laundering. Quarpel, however, has given a truly permanent water repellent finish to cotton fabrics. On sateen, it is as good after 15 launderings as a new pyridinium type finish.

Laxer: You say that the odor from durable creasing with monoethanolamine sulfite is objectionable. We never have complaints about this. Army men must have unusually sensitive noses.

Kennedy: If you were on the receiving end of complaints from high-ranking officers, your nose would become sensitive, too. We have noted an odor when the item becomes wet.

Stitt: Isn't there some confusion on the question whether high moisture regain is or is not desirable for body insulation?

Kennedy: We are changing our views on this subject. Under conditions such that sorbed moisture is repeatedly evaporated by body warmth and condensed by environmental cold, the attendant extra heat loss is undesirable and may be serious. For this reason Paul Siple designed, for Arctic use, boots with sealed insulation. This consideration is especially important for feet, which must be protected from both condensed moisture and excessive heat loss.

Lundgren: What is the incidence of allergy due to wool?

Kennedy: We have no data. There are some complaints, but we feel that the incidence of any important distress is very low.

Laxer: What has happened to the Army use of blankets?

Kennedy: We have stopped issuing blankets for field use because they are less satisfactory than feather sleeping bags. However, blankets are still used in barracks.

Klemer: Are you concerned with bactericidal finishing treatments?

Kennedy: We would view a durable bactericidal finish with great interest.

THE DEVELOPMENT OF EASY-CARE PROPERTIES FOR WOOL FOR CIVILIAN USE

Gerald Laxer
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(Dr. Laxer was originally scheduled to discuss consumer preferences in wool. However, he remarked that these were well documented, so that he felt that it would be more valuable to consider the nature of easy-care requirements that may affect consumer preferences in the future.)

Some kinds of wool garments are well known to be successfully launderable under proper conditions. To define the required garment properties and laundering conditions, the Wool Bureau, for example, has supported research by the Harris Research Laboratories on effects of fiber, yarn, and fabric construction and of laundering conditions on launderability. ("Structural Compactness of Woven Wool Fabrics and Their Behavior in Modern Washing Machines," H. Bogaty, G. H. Lourigan, and H. E. Harris, Textile Res. Jour. 28: 733, 1958; "Some Factors Involved in 'Wash-and-Wear' Wool," J. F. Krasny, H. E. Harris, G. H. Lourigan, and E. D. White, Amer. Dyestuff Reptr. 48: 31, 1959; "Progress in 'Wash-and-Wear' Wool," J. F. Krasny and J. Menkart, Amer. Dyestuff Reptr. 48: 114, 1959.)

These and similar studies show that garments suitably made from compactly woven material will launder without excessive shrinkage when "mild" washing cycles of modern home washing machines are used. Furthermore, washing machine manufacturers express willingness to include special cycles for woolens if this should prove desirable. In addition to dimensional stability, the wash-and-wear idea implies tumble-drying without excessive fuzziness or mussiness. Summarizing, commercial wool garments can be made regularly available that may properly be considered wash-and-wear products when machine laundered with washing and drying cycles adapted for synthetics, including use of small loads, low agitation, and minimum soaping cycle.

Properties of wool fabrics that favor easy care are use of fine yarn with relatively high twist and a compact weave. Heavier fabrics show the best muss resistance. Failure in laundering is commonly due to seam puckering or to poor dye fastness. Tumble-drying causes shrinkage only if the fabric tends to shrink also in the wash cycle. Tumbling, as opposed to drip-drying, gives better smoothness, but more fuzz, but fuzz is minimized by satisfactory shrinkage control. With the more open fabrics, chemical treatment can be used to achieve satisfactory wash-and-wear performance.

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* The Wool Bureau, Inc., is a nonprofit organization for promoting use of wool in the United States. Wool growers of Australia, South Africa, and New Zealand support it. It operates by publicity, advertising, and merchandising, and sponsors research on fundamental wool properties, processing, and marketing.

To show a trend, current newspaper advertising of permanent-creased men's suits was mentioned. Durable crease finishing is now used by nearly 50 major garment manufacturers in the United States and Canada. These are convinced that durable creasing will be used more and more routinely. Manufacturers will use new processes when they are convinced that better products result. Technical details of use of monoethanolamine sulfite for durable creasing of all wool trousers were shown in a movie. (Technical details and film are available through the Wool Bureau.) Much promotion and liaison work is needed to get even a good process out of the laboratory and into commercial use. Liaison is especially critical for natural fibers.

Discussion of Dr. Laxer's Report

Question: Will you comment on laundering and softening agents suitable for home laundering of woolens?

Laxer: The most important conditions have been mentioned. Wool should not be washed in very alkaline media. The commonly available nonionic detergents should be satisfactory with any public water supply. Use of cationic softeners is a matter of personal preference. In any case, control of agitation is the most important requirement.

Question: What are the relative merits of the use of monoethanolamine sulfite and of monoethanolamine as the free amine, in water solution, as suggested by the Department of Agriculture? (News release USDA 1974-60 for release July 15, 1960.)

Rosenstein: Kuppenheimer has tested both and is still using the sulfite.

Lundgren: There is some question about the mechanism of the treatment since the amine itself is not a reducing agent. It may promote formation of lanthionine residues, which are assumed to be more stable than the original cystine residues. Use of the free amine avoids the problem of off odor of the wet fabric.

Question: How does the drycleaner put the crease back in the same place in durably creased trousers?

Laxer: He doesn't always. This requires attention. But if he makes a mistake, the second crease isn't durable and can be corrected by pressing again.

Question: If the crease is durable, why does the label say to dryclean the garment?

Rosenstein: Drycleaning protects the trim materials. More important, the durable-creasing treatment does not give protection against felting shrinkage.

Question: What effect does the durable-creasing treatment have on abrasion resistance?

Laxer: We haven't wear-tested durable-creased goods. The consensus from the literature is that wearing properties are not appreciably affected. However,

there have been complaints of creases breaking. This suggests that further attention should be given to the fiber content and fabric construction.

Du Pre: Is the ethanolamine sulfite process effective for wool blends?

Laxer: Yes, except that the treatment as described does not set the synthetic component. It would be necessary to use a higher temperature to set Dacron, for example.

Copley: What improved properties does wool need to regain markets that it has lost?

Laxer: Considering wool-Dacron blends, these have taken over the lightweight suiting market because even with the best construction, wrinkle resistance and recovery from wrinkling at high relative humidity and temperature are better for the blend than for all-wool fabric. The weight of the fabric is also important. Furthermore, there are manufacturing advantages. For the fine yarns that are required, Dacron content materially decreases breakage in spinning and weaving. These facts make it probable that blends in this case are here to stay.

Burns: Would it be feasible to make laboratory frock coats out of light weight woolen or worsted?

Laxer: Such coats would no doubt cost more than comparable cotton ones.

WHAT'S NEW IN COTTON CHEMICAL FINISHING AT SU

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(Some recent developments in the following areas of chemical finishing were discussed: Flame resistance, rot and mildew resistance, wash-wear, and all-cotton stretch yarns and fabrics. Chemical substances and reactions mentioned are shown in Table 1.)

Chemical modifications. The reaction of chloromethylphosphonic acid with cotton in the presence of sodium hydroxide represents a typical etherification reaction with cellulose. This particular reaction goes quite readily. At low degrees of substitution (DS), cotton cellulose becomes hydrophilic but retains its fibrous structure; at high degrees of substitution, about DS 0.2, the modified cotton becomes soluble in dilute alkali. Chemical modifications such as this modify both the chemical and physical properties of cotton. A use for soluble cotton can be exemplified with carboxymethylated cotton. A lace pattern is woven on top of soluble carboxymethylated cotton. Afterwards the soluble cotton is dissolved, leaving a very delicate lace which could not have been produced without the use of a soluble fiber.

Flame-resistant cotton. The Southern Regional Research Laboratory has developed about six durable flame-resistant finishes for cotton. All of these finishes are quite good, but one of these, referred to as APO-THPC, is outstanding. The two main chemicals used in this finish are APO and THPC (see Table 1). Both can react with cotton as well as co-react to form a polymer. The flame-resistant finish is applied to cotton by first dissolving these chemicals in water and padding them on the cotton fabric. Afterwards, the fabric is heated to about 140° C. for a few minutes to promote reaction with the cotton and polymerization. The finish produced is extremely resistant to removal by laundering and drycleaning. This finish is effective on 8-ounce cotton fabrics with about 10 percent add-on. It is applicable to lightweight as well as to heavier cotton fabrics.

Rot and mildew resistance. For a number of industrial uses, cotton must be resistant to rot and mildew and destruction by sunlight. A recent finish developed at the Southern laboratory is a very effective finish of this type. It is called the acid-colloid finish. It is produced by dissolving trimethylolmelamine and formic acid in water, padding cotton fabric with this solution, and then curing at about 140° C. It is necessary to use about five moles of formic acid per mole of trimethylolmelamine. This finish penetrates cotton fibers to a limited extent. Most of the polymeric material is found in the outermost part of the secondary wall of the cotton fibers. Treated cotton products are extremely resistant to rot and mildew. A treated cotton fabric will retain nearly 100 percent of its strength when placed in a soil bed for 21 weeks, whereas an untreated cotton fabric will be essentially destroyed in four days. Acid-colloid-treated cotton fabrics have about twice the resistance to actinic breakdown as untreated cotton fabrics.

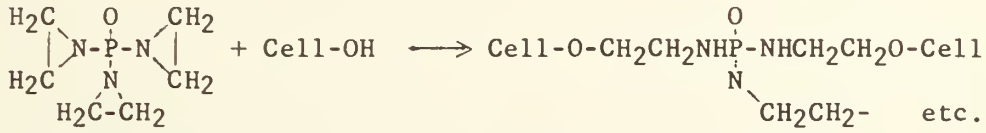
Table 1. Formulas and reactions of cotton finishing chemicals.

Cotton cellulose is represented by Cell-OH, showing a representative reactive hydroxyl group. When the product is shown with two "Cell" groups, this shows that the reagent can react with another hydroxyl group on the same or a different cellulose chain, permitting cross-linking. In some cases the reagent can also react with itself or in other ways, giving more complex products than indicated.

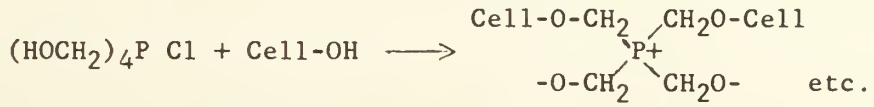
1. Chloromethylphosphonic acid

$$ClCH_2PO(OH)_2 + \text{Cell-OH} \longrightarrow \text{Cell-O-}CH_2PO(OH)_2$$

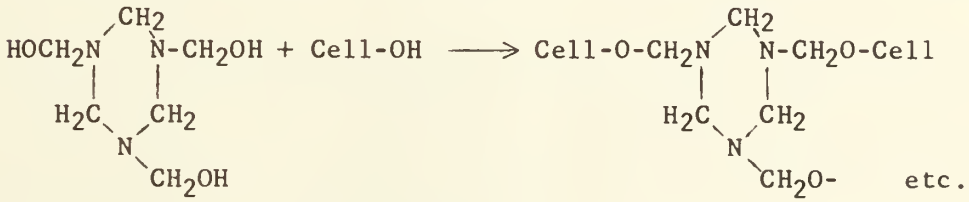
2. APO Tris(1-aziridinyl)phosphine oxide



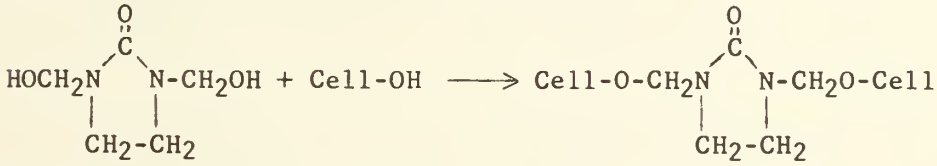
3. THPC Tetrakis(hydroxymethyl)phosphonium chloride



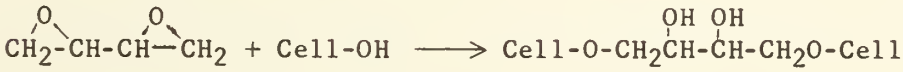
4. Trimethylolmelamine (used with formic acid in the "acid-colloid" process)



5. Dimethylol cyclic ethylene urea



6. Butadiene diepoxide



7. Formaldehyde

$$H_2CO + \text{Cell-OH} \longrightarrow \text{Cell-O-}CH_2O\text{-Cell}$$

Wash-wear. It has been well demonstrated that it is necessary to crosslink the cotton cellulose molecules in order to produce wash-wear properties. Cotton that has reacted with a monofunctional reagent, such as N-methylol-N'-methyl-cyclic-ethyleneurea, does not have any appreciable degree of wrinkle resistance. On the other hand, cotton treated with a polyfunctional reagent, such as N,N'-dimethylol-cyclic-ethyleneurea, has a very high degree of wrinkle resistance. The compound APO, which is trifunctional with respect to cotton cellulose, crosslinks cotton to produce a very high degree of wrinkle resistance. An acid catalyst, such as zinc fluoborate, is normally used to catalyze the reaction of APO with cotton. This finish is very durable to all of the usual types of laundering and drycleaning. Fabrics treated with this compound turn yellow when bleached with sodium hypochlorite; thus the finish is not suitable for white fabrics, although it should be excellent for colored goods.

Butadiene diepoxide appears to be an extremely efficient crosslinking agent for cotton. A very high degree of wrinkle resistance is obtained with about 1 percent add-on of this reagent. One percent of this reagent represents a degree of substitution of only 0.02 percent. A DS of 0.05 is required with most other reagents examined. It may be that this reagent crosslinks in a particularly effective area of the cotton fiber, or it may be that all of the molecules, or nearly all, are used in intermolecular crosslinking, rather than intramolecular reaction.

Formaldehyde is cheap and will crosslink cotton. Since it is such an attractive reagent for finishing cotton, considerable effort is being placed upon this reagent. Studies up to now have shown that wet-wrinkle resistance can be obtained by crosslinking with formaldehyde while the cotton fiber is in a highly swelled condition, as when swelled in water. Wet- and dry-wrinkle resistance are obtained when the cotton fiber is crosslinked in a relatively nonswelled condition. This has been accomplished by immersing cotton fabrics in a solution containing a very high percentage of acetic acid and small amounts of hydrochloric acid, formaldehyde, and water. Wet- and dry-wrinkle resistance has also been obtained by immersing cotton fabric in an aqueous solution containing 35 percent calcium chloride, formaldehyde, and hydrochloric acid. The Southern laboratory is also exploring possibilities of using formaldehyde gas for the treatment of cotton. In this case, the air-equilibrated cotton fabric is merely exposed to an atmosphere containing formaldehyde and a trace of hydrochloric acid. Wet- and dry-wrinkle resistance are obtained under these conditions also.

All-cotton stretch yarn and fabrics. Two general approaches are being made to develop all-cotton stretch yarns and fabrics for apparel use. One approach is based upon the use of crosslinking agents of the same types that are used for the production of wash-wear cottons. In this approach, the general process consists in impregnating the cotton yarns with the crosslinking agents, giving it an overtwist, then curing the resin, and afterwards backtwisting slightly to the desired amount. The resin or crosslinking agent sets the yarns so that the backtwisting produces bulk and stretch in the yarn. This research has proceeded to the state where socks have been made. They appear quite attractive but have not been evaluated as yet.

The second approach being used for the development of all-cotton stretch yarns and fabrics is based upon the development of thermoplastic cotton. This is done by treating cotton with monofunctional reagents that will introduce hydrophobic side groups. This generally takes a degree of substitution in the order of about 0.5 to 1.0. For example, cotton is made thermoplastic by acetylation to a DS of 1, or by benzoylation to a DS of about 0.5. One way of producing stretch yarn from these thermoplastic fibers is to overtwist yarn and then heat above the second order transition temperature for a very brief period while the yarn is overtwisted, then backtwist slightly when the yarn has cooled. This produces a crimped yarn which, when knitted into fabric, has a wool-like feel. The stretch appears to be durable.

Discussion of Dr. Reeves' Report

Question: Can the APO-THPC flame retardant treatment be used on wool and other fibers?

Reeves: This flame retardant performs best on cotton but can be used to flame-proof other natural fibers such as silk. It is also effective on other cellulosic fibers such as rayon. It does not work on hydrophobic fibers, such as Dacron and Nylon, except when these fibers are blended with cotton and there is at least 50 percent cotton in blend.

Question: How much strength is retained by fabrics treated with the various formaldehyde processes?

Reeves: Where only wet-wrinkle resistance is obtained, the tear and tensile strengths are generally as good as, and sometimes better than, is obtained with the conventional resins. In all cases, the best strengths are obtained with mercerized fabrics. In the processes using acetic acid or calcium chloride solutions, the strengths retained were, in some cases, as good as those obtained with conventional resins; in other cases, somewhat less. There are unknown factors that affect the strength of fabrics treated by either the calcium chloride or acetic acid process. It appears that the strength is lowered substantially when the formaldehyde reacts with cotton in the vapor phase. This study has just begun; it is too early to predict what may be obtained through this process.

SOME ASPECTS OF WOOL FINISHING

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Mr. von Bergen began by calling attention to the microscopic details of wool fiber structure including, particularly, the now well-known differences in chemical reactivity, including swelling and dyeing, between opposite sides of a typical fine wool fiber. He then reemphasized that Orlon 21 has been made with a double structure giving it a crimp in three dimensions, simulating further a desirable property of wool.

Work of D. L. Munden of Leeds University was cited in which the shrinkage of knitted fabric was illustrated by yarn behavior. ("Dimensional

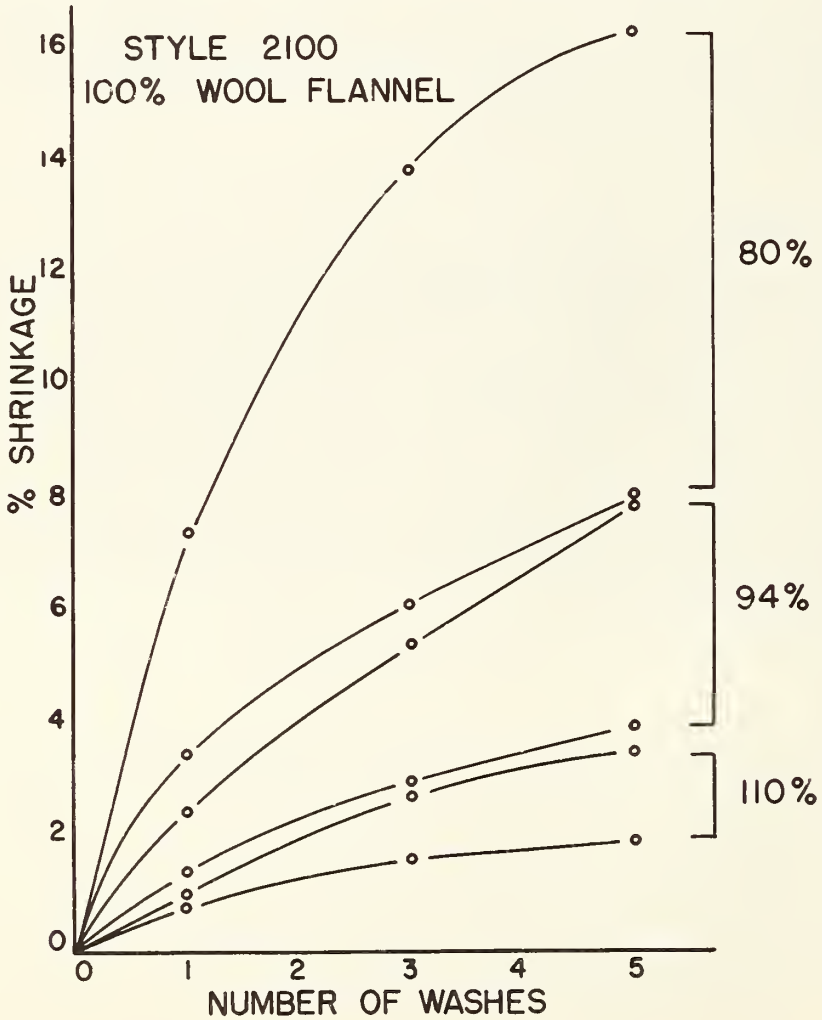

Figure 1. The influence of the cover factor (shown at the right, %) on the shrinkage of all-wool flannel in machine laundering.

Stability of Plain-Knit Fabrics," D. L. Munden. Jour. Textile Inst. 51: P200, 1960. cf. Table I.) This shows very strikingly that, in addition to felting shrinkage, wet relaxation and consolidation must be taken into account.

Fabric construction is critically important for dimensional stability. To illustrate this, the progressive shrinkage of flannels of various cover factors was shown resulting from a series of machine launderings (Figure 1). Fabric with 80 percent coverage shrank greatly, whereas that with 110 percent coverage shrank only 2-1/2 percent in the warp direction, 1 percent in the fill direction. However, the latter (15-oz.) fabric was much too heavy for most practical uses. Considering use of these materials for skirts, it was noted that chemical treatment such as chlorination considerably reduces shrinkage, but that the untreated fabric with 110 percent coverage performed better than the treated fabric with 80 percent coverage. At present it is not economically feasible to use such tight construction as the principal means of shrinkage control.

Napping was discussed as an important finishing step which merits more attention in wool finishing. Representative napped wool fabrics are suede, in which the nap is very short and dense, velour, with a nap 1/8 to at most 1/4 inch, and fleece, with a nap up to about 1/2 inch. One value of napping is that it improves the water-shedding properties of the fabric. Artificial furs are made from Orlon fabrics, which are made with a very long nap and high luster. The fibers of the nap are aligned and straightened by heat and pressure in the same sort of machinery used with sheepskins for the production of mouton furs and now also by drycleaners for treating napped materials. The essential part of such machinery is a heated polished cylinder with oblique grooves, which are of various forms. An example illustrated was the Electro-finisher made by the Turbo Machine Company. The main problem in napping is that of permanence. In general, the fibers relax when the garment is steamed (or even when wet) and the luster disappears. Many processes have been proposed to achieve permanence, using such reagents as formaldehyde, formic acid, and alcohol. J. B. Speakman has proposed use of bisulfite with ethylene glycol ("The Permanent Lustring of All-Wool Fabrics," J. B. Speakman and M. A. Wolfram, Jour. Textile Inst. 49: T627, 1958). None of these processes appears fully satisfactory. There is still important research to be done on this subject.

A further very difficult problem faced by light-weight wool garments is the change in shape and surface smoothness caused by changes in relative humidity ("Dimensional Stability of Woolen and Worsted Fabrics," W. von Bergen and C. S. Clutz. ASTM Bulletin No. 167, 74, July 1950). The effect is very striking (Figures 2 and 3), so much so that suits made up expertly in New York at 20 to 30 percent relative humidity may be judged completely unsuitable on arrival in Los Angeles at 90 percent relative humidity. Gabardine garments are especially sensitive, but tropical worsteds are also seriously affected.

A most important observation is that the adverse changes due to humidity changes can be prevented almost entirely by use of a blend with 25 percent Dacron. Considering this fact, it is almost foolish not to use man-made fibers to make a better wool product.

Figure 2. Movement of fabrics with relative humidity; change in the appearance of a light-weight suit coat with change in relative humidity.

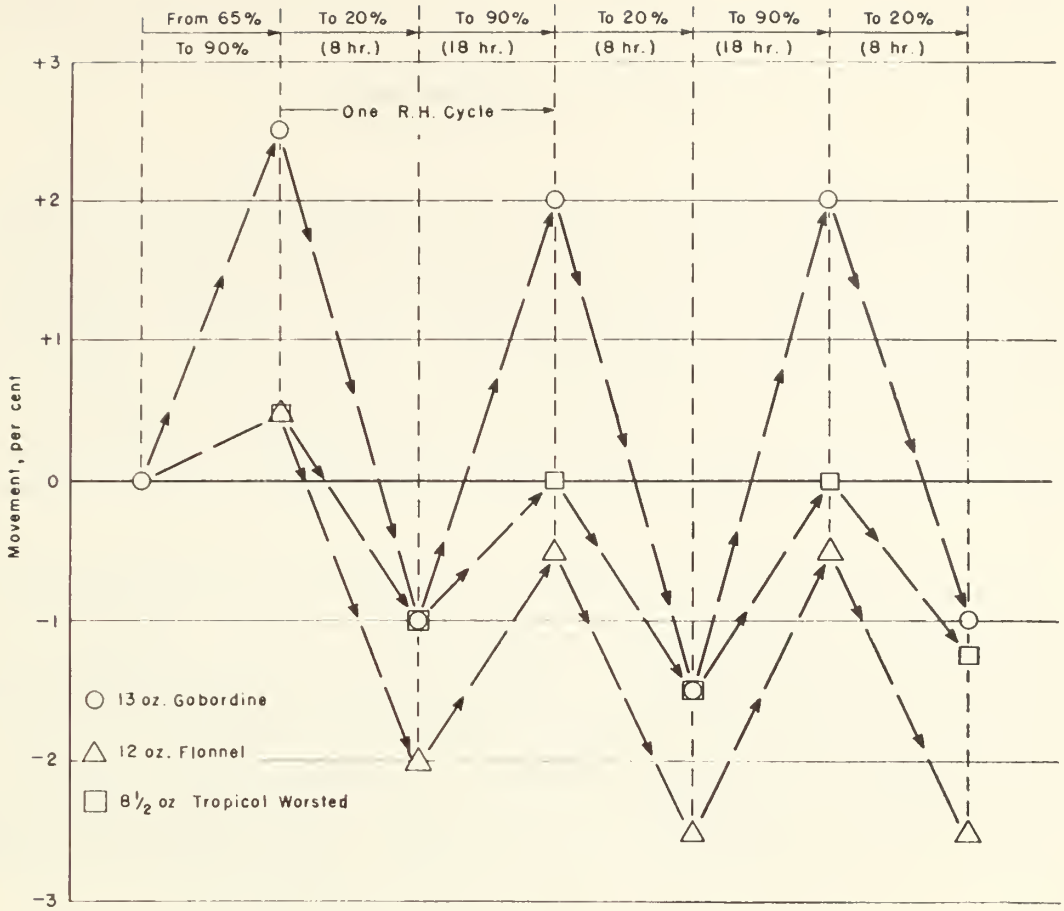

Figure 3. Movement of fabrics with relative humidity measured in warp direction with material lying flat and at 70° F.

This consideration may be extended to other products, which, for specific uses, may possibly be improved by incorporating appropriate amounts of synthetic fibers. In this endeavor the initiative has been entirely with the manufacturer interested in promoting his fibers. ("Properties of Blended Fabrics," Du Pont Bulletin X21, Dec. 1954. E. I. Du Pont de Nemours and Co., Wilmington, Del.) The time has come for an objective appraisal of the properties of blends to be sure that the proportions proposed for a given use are reasonable.

Discussion of Mr. von Bergen's Report

Lundgren: In some wool-synthetic blends, repeated drycleaning is reported to bring the synthetic component to the surface. Is this an important problem?

von Bergen: It is a problem especially noticeable in cross-dyed materials with the synthetic component undyed; less so in solid shades. As far as I know there are very few consumer complaints. Pilling is a more important problem with blends.

HOW BASIC RESEARCH IS AIMED TOWARD ACHIEVING NEW AND BETTER FINISHES

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This conference has emphasized practical problems of wool finishing. Research on these problems has been reported with special attention to developments thought to have most immediate practical value. However, with proper concern for the practical, it is appropriate to note that these developments are supported by a continuing program of basic research attacking many areas of ignorance in wool, that most complicated of all textile fibers. Technical progress depends very directly on the encouragement of basic research, which, although it should have well defined goals, nevertheless typically leads to unlooked-for results and implications that may be of great practical value to the alert.

Basic research on wool and mohair is concerned with structure of fibers at all levels of organization, with composition and chemical reactivity, and with physical properties and energy relationships. Within this broad scope are many challenging areas of ignorance. These often overlap. Mechanical and other physical properties have not been accounted for in detail. For example the processes of water sorption and the state of water held in wool still present unsolved problems. Many important relationships among composition, structure, and physical properties have not been determined for lack of primary data in each category. Although the average amino acid composition of wools is well established, specific larger components have not been well characterized or related to structure except in a very rudimentary way.

Procedures for specific and efficient chemical modification are commonly unavailable, or their applicability to wool is not critically evaluated. Detailed mechanisms of degradation by chemical attack, light, and heat are largely elementary and speculative. Even very direct practical needs are unsatisfied for want of fundamental techniques or insight. For example, quick, convenient, objective tests of wool quality are still being sought. Processes leading to felting shrinkage are still not understood in detail. Mechanisms of failure of wool in use are mostly unknown.

Studies of such fundamentals will not inevitably lead to better finishing treatments or other technical or economic improvements, but they do provide necessary information and insight not otherwise available. Practical developments require in addition imagination and knowledge of existing technical requirements and possibilities.

In practice it may be impossible to classify specific research as fundamental or applied. The classification depends in part on point of view. However, some research activities here that are of basic interest may be noted. First, methods are being explored and developed for chemically modifying wool in well defined ways, either by making simple changes at specific reactive sites or by incorporating polymeric material. Comparison of specifically

modified and natural wools is fundamental to the whole of wool research. It may be remarked that such activity has led to several developments of practical promise, including those reported in detail to this conference.

Second, attempts are being made to degrade and modify wool systematically to obtain soluble constituents that can be separated, analyzed, characterized in detail, and finally related to fiber structure and properties. Acrylonitrile has been used to make such soluble derivatives. High-molecular-weight fractions have been obtained that differ in composition from the original wool, but have recently been found to have several discrete components. The basic questions are: "How are these derived chemically from the original fiber?" and "Where do they come from in the original structure?"

Mechanisms of degradation by ultraviolet light are being studied by amino acid analysis, by mass spectrometry, by optical measurements, and by observation of electron paramagnetic resonance. These methods complement one another in showing the relative sensitivity of the various amino acid residues to attack, the identity of volatile products formed under various conditions, and the nature of the first observable products of irradiation.

Such studies are typical of the utility of appropriate physical methods including optical and electron microscopy, X-ray diffraction, and infrared, visible, and ultraviolet spectroscopy, among others, both to define and to guide chemical treatment and to provide details of wool structure and structural changes. Nuclear magnetic resonance spectroscopy has been especially valuable in determining the interaction of hydrogen atoms with their immediate environments. By this means, further details of adsorption and desorption of water are being determined.

Mechanical properties of natural and modified single fibers receive continuing attention, both stress-strain measurements, for which automatic computation of results is expected to be especially helpful and also measurements of properties, such as the torsional modulus, that are more sensitive to changes in surface structure.

Finally, mechanical properties of fiber assemblies and fabric are evaluated routinely, with continuing effort to develop objective measurements related to the more subtle textile qualities such as hand, drape, and smoothness. Although these measurements directly concern practical properties, they also serve to establish basic relations between textile qualities and the physical and chemical properties of the constituent fibers.

To summarize, practical problems, such as protection against shrinkage, characteristically receive both trial-and-error attempts at solutions and studies aimed primarily at understanding. The two activities are not mutually exclusive, but support one another and may merge. In the case of shrinkage, it may be that development of feasible means of control has actually outstripped detailed understanding of the essentials.

TOUR OF BASIC RESEARCH LABORATORIES

The conference concluded with a tour of selected basic research facilities.

Richard S. Thomas showed an electron microscope and associated equipment for electron microscopy of fibers and other agricultural materials. Electron microscopy is being used initially to determine surface changes in resin-finished wool fibers.

Herbert A. Launer showed equipment used for ultraviolet irradiation of wool with light of known spectral distribution and intensity and at controlled temperature. William H. McFadden demonstrated a mass spectrometer, with associated equipment as used for analyzing gaseous decomposition products of wool exposed to ultraviolet light or heat.

Robert E. Lundin and Richard H. Elsken demonstrated a 60-megacycle, dual purpose nuclear magnetic resonance spectrometer, with 12-inch magnet and other associated equipment, widely used for analysis of the structure of organic materials in terms of the environments of their hydrogen atoms. This instrument is being applied to the study of details of adsorption and desorption of water from wool.

John J. Windle demonstrated an electron paramagnetic resonance spectrometer (sometimes described as electron spin resonance spectrometer) based on a 6-inch electromagnet and associated equipment. This is indispensable for studying unpaired electrons due to free radicals produced in wool by irradiation.

Dale R. Black explained the principles of X-ray diffraction analysis applied to wool and chemically modified wool. Equipment used for this research consists of vacuum cameras, low-angle and normal cameras designed and built here, together with various accessories including a diffractometer, used particularly for study of crystalline materials.

Walter J. Thorsen, with technical assistance of Helen Kivela, is responsible for amino acid analysis of protein hydrolyzates. Such analyses are fundamental to the study of fiber components as well as to study of changes in composition due to chemical modification or physical degradation. An amino acid analyzer used for this work was shown in operation.

A fabric and fiber sound spectrum analyzer engineered and built commercially from specifications developed here was demonstrated with associated apparatus as used for studying the hand and wrinkledness of fabrics. In addition, Mr. Thorsen displayed the electric analog computer, with accessories arranged to display significant stress-strain parameters from single-fiber tests made with a tensile tester with wide column separation.

Finally, Richard A. O'Connell and Raymond J. Martsch demonstrated a particle size counter. This device determines the size and size distribution of particles suspended in a liquid. It can be used to determine fiber fineness and fineness distribution in wool top. Conditions are being sought that will permit it to be used reliably and conveniently on a routine basis as an improvement over the present standard microscopic procedure.

The Wool and Mohair Laboratory also has facilities in use for optical microscopy, for spectroscopic analysis of emission spectra of metals, of reflected light, and of transmitted light in the visible, infrared, and ultraviolet ranges, for electrophoresis, and for the study of molecular weight by ultracentrifugation and by light scattering.

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ACKNOWLEDGMENT

In the preparation of this report, Wilfred H. Ward, chemist, Wool and Mohair Laboratory, reconstructed the substance of the discussions, prepared summaries of most of the papers, and described the demonstrations and tour. His important contribution is gratefully acknowledged by those who presented information.

GPO 982177

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